Maintaining your Agilent GC and GC/MS Systems

Our measure is your success.

Agilent Technologies

Table of Contents

Gas Management 1

Gas Types 2
Gas Purification Systems 4
Gas Traps 6
Renewable Gas Purification System 12
Universal/External Split Vent Trap 13
Regulators 14
Tubing 15
Flowmeters 16
Gas Leak Detector 18

Sample Introduction 19

Vials 20
Electronic Crimpers and Decappers 25
Gold Standard Autosampler Syringes 26
Headspace Sampler 34
CTC Autosampler Supplies 37
Purge and Trap Supplies 41
Markes Thermal Desorption 43

Inlets 45

Premium Non-Stick Septa 46
Ferrules 50
Capillary Flow Technology Supplies 54
Split/Splitless Liners 55
Direct Connect Liners 59
Helix Liners 59
Split/Splitless Inlets 60
Cool On-Column Inlets 69
Purged Packed Inlets 75
Programmed Temperature Vaporizer (PTV) Inlets 78

Detectors 83

Flame Ionization Detector (FID) 84
Electron Capture Detector (ECD) 91
Thermal Conductivity Detector (TCD) 94
Flame Photometric Detector (FPD) 99
Nitrogen Phosphorus Detector (NPD) 106
Nitrogen and Sulfur Chemiluminescence Detectors 113

GC/MS Systems 115

Maintaining Mass Selective Detectors (MSD) 116
MSD Contamination 118
Cleaning and Maintenance Supplies 126
Ion Source 128
Installing a Capillary Column in the GC/MSD Interface 134
QuickSwap MS Interface Restrictors 135
MSD Filaments 136
Vacuum Systems and Pumps 138
MSD Electron Multipliers and Replacement Horn 142
MS Test and Performance Samples 143

Gas Management

Avoid column damage, improve detector life, and achieve reliable, consistent results.

Gas management means more than just selecting the appropriate carrier gas for your needs. It also means taking steps to prevent contaminants like oxygen, moisture, and hydrocarbons from damaging your GC column.

As you read through the following pages, you will find...

- Practical advice about carrier gas types, impurities, leak detection, and flow rates.
- Valuable techniques that can minimize the likelihood of GC system contamination.
- Tips and tools that will help your lab implement gas management protocols.
- The latest details about Agilent-engineered gas purifiers, tubing, regulators, leak detectors, and flowmeters.

Gas Types

Carrier Gases

The most frequently used carrier gases are helium and hydrogen, although nitrogen and argon can be used. Purity is essential for these gases since they sweep the sample through the column where it is separated into its component parts and then through the detector for component quantification. Carrier gas purity is also critical to prevent degradation of chromatographic columns and hardware.

The following tables list minimum and maximum pressures in psi for inlets and detectors measured at the bulkhead fitting at the back of the gas chromatograph.

Inlet Pressure Settings (psi)

	Split/Splitless (0-150 psi)	Split/Splitless (0-100 psi)	Cool On-Column	Packed	PTV
Carrier max (psi)	170	120	120	120	120
Carrier min (psi)	Must supply 20 psi greater than pressure used in method				

Detector Pressure Settings (psi)

	FID	NPD	TCD	ECD	FPD
Hydrogen	35-100	35-100			45-100
Air	55-100	55-100			100-120
Make up	55-100	55-100	55-100	55-100	55-100
Reference			55-100		

Conversions: 1 psi = 6.8947, kPa = 0.068947, Bar = 0.068 ATM

Gas Purity and Selection

Agilent recommends that carrier and detector gases be 99.9995% pure. Air needs to be zero grade or better. Agilent also recommends using traps to remove hydrocarbons, water and oxygen.

When used with capillary columns, GC detectors require a separate makeup gas for optimum sensitivity. For each detector and carrier gas, there is a preferred choice for makeup gas. This table lists gas recommendations for capillary columns.

Detector	Carrier Gas	Make up 1st choice	Make up 2nd choice	Purge or Reference
Electron capture	Hydrogen	Argon/methane	Nitrogen	Anode purge must be same as makeup
	Helium	Argon/methane	Nitrogen	
	Nitrogen	Nitrogen	Argon/methane	
	Argon/methane	Argon/methane	Nitrogen	
Flame ionization	Hydrogen	Nitrogen	Helium	Hydrogen and air for detector
	Helium	Nitrogen	Helium	
	Nitrogen	Nitrogen	Helium	
Flame photometric	Hydrogen	Nitrogen	None	Hydrogen and air for detector
	Helium	Nitrogen		
	Nitrogen	Nitrogen		
	Argon	Nitrogen		
Mass selective	Hydrogen	None	None	
	Helium	None	None	
Nitrogen phosphorous	Helium	Nitrogen	Helium	Hydrogen and air for detector
	Nitrogen	Nitrogen	Helium	
Thermal conductivity	Hydrogen	Must be same as carrier and reference	Must be same as carrier and reference	Reference must be same as carrier and makeup
	Helium			
	Nitrogen			

Contaminants & Purities

Contaminants in gases are major contributors to capillary column degradation and detector noise, and can interfere with chromatographic results. Concentration of these contaminants vary by the grade of gas.

Hydrocarbons and Halocarbons

- Decrease detector sensitivity by increasing detector background noise.
- Can also cause baseline drift or wander, contaminant peaks, and noisy or high offsets of baselines.

Moisture

- Can be introduced by improper handling and/or installation of plumbing.
- A common cause of column stationary phase degradation.
- Can damage instrument.

Oxygen

- Most common contaminant.
- A common cause of column stationary phase and inlet liner degradation.
- Can cause decomposition of labile analytes.
- Opportunity for introduction at every fitting present in the gas line or during use of gas permeable tubing.

Tips & Tools

Gas purifiers should always be oriented vertically, not horizontally.

Gas Purification Systems

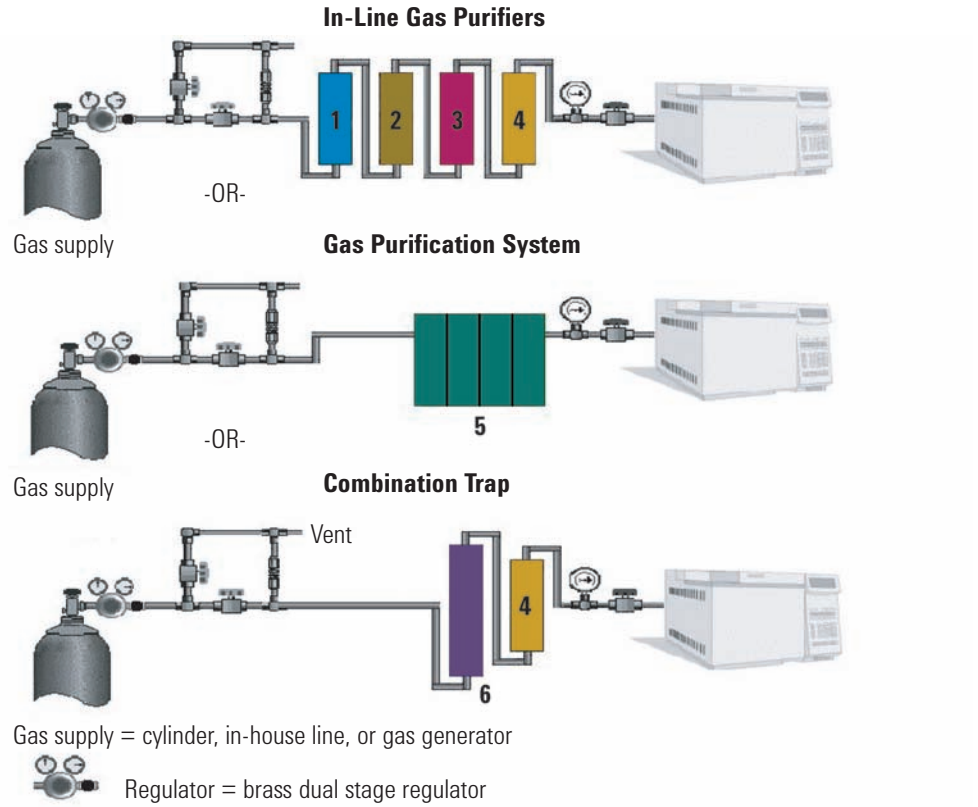
Purifiers are available in a variety of sizes and configurations to remove common contaminants like oxygen, moisture, and hydrocarbons. In-line gas purifiers, including refillable, indicating, S-shaped, and metal body types, are made to remove specific contaminants. Agilent also offers gas purification systems with removable cartridges. These systems provide the ability to design the right combination of filters needed for your application to achieve the proper gas purity.

Carrier Gas Purification

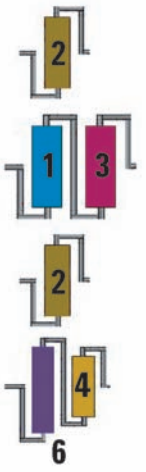
The Carrier Gas Purification illustration shows the most common gas purification configurations used in gas chromatography.

Regardless of which purification system is employed, proper installation and maintenance is required to achieve optimal performance from the purification system(s). A purifier that is not maintained will eventually expire and become ineffective, or worse, a source of contamination.

- Determine desired purity level.
- Keep number of fittings in gas line to a minimum.
- Install purifiers in a convenient location close to the GC.
- Purifier log books are useful for determining maintenance schedule.
- Use indicating traps closest to the GC so you can determine when to change the traps that are upstream.



Detector Gas Purification



FID make-up, air, and H_2

ECD make-up

ELCD reaction gas

MS carrier gas

Key

1 = Moisture Trap

2 = Hydrocarbon Trap

3 = Oxygen Trap

4 = Indicating Oxygen Trap

5 = Gas Purification System

6 = Combination Trap for moisture, oxygen, and hydrocarbon removal

Gas Traps

The purpose of gas traps is to remove detrimental impurities from the carrier and detector gases. Combination traps are available which remove moisture, oxygen and/or organics with a single trap. The effectiveness of the traps depends on the initial quality of the gas.

Constant exposure of capillary columns to oxygen and moisture, especially at high temperatures, results in rapid and severe column damage. The use of oxygen and moisture traps for the carrier gas may extend column life and protect the instrument. Any moisture or oxygen introduced into the gas stream due to a leak will be removed by the trap until it expires.

Moisture (Water) Traps

Indicating moisture traps are available in plastic and glass bodies. Glass body traps are used when potential contaminants from plastic trap bodies are a concern. Glass traps are normally encased in a protective, plastic shrink wrap or a high impact plastic shield (outer trap body). Glass and plastic bodied traps are usually pressure tested at 150 psi, thus they are safe for use at the typical pressures required by the GC.

Moisture trap

Moisture Traps

Description	Size (cc)	Removal Capacity (g)	Max. Effluent H_2O Concentration (ppb)	1/8 in. Part No.	1/4 in. Part No.
Molecular Sieve 13X and Indicating 4A (MT Series)					
Refillable Moisture Trap	120	21.6	20	MT120-2	MT120-4
Refillable Moisture Trap	200	36.0	18	MT200-2	MT200-4
Refillable Moisture Trap	400	72.0	14	MT400-2	MT400-4
Adsorbent refill (1 pint) for MT series				MSR-1	MSR-1
Molecular Sieve 5A and Indicating Drierite (MT-D Series)					
Refillable Moisture Trap	120	21.6	22	MT120-2-D	MT120-4-D
Refillable Moisture Trap	200	36.0	20	MT200-2-D	MT200-4-D
Refillable Moisture Trap	400	72.0	16	MT400-2-D	MT400-4-D
Adsorbent Refill (1 pint) for MT-D Series				MSR-2	MSR-2

Tips & Tools

Moisture traps are highly recommended for carrier and ECD gases.

Moisture Traps

Description	Size (cc)	Removal Capacity (g)	Max. Effluent H_2O Concentration (ppb)	1/8 in. Part No.	1/4 in. Part No.
Silica Gel, Grade 40, and Indicating Silica Gel, Grade 48 (MT-S Series)					
Refillable Moisture Trap	120	31.5	40	MT120-2-S	MT120-4-S
Refillable Moisture Trap	200	52.5	39	MT200-2-S	MT200-4-S
Refillable Moisture Trap	400	105.0	39	MT400-2-S	MT400-4-S
Adsorbent Refill (1 pint) for MT-S series				SGR	SGR
Glass Indicating Moisture Traps (GMT and LGMT Series)					
Glass Indicating Moisture Trap	70	11.4	7	GMT-2GC-HP	GMT-4GC-HP
Glass Indicating Moisture Trap	100	16.3	6	GMT-2-HP	GMT-4-HP
Glass Indicating Moisture Trap	250	40.09	6	LGMT-2-HP	LGMT-4-HP
Molecular Sieve Refill for GMT and LGMT series				GMSR	GMSR
Moisture Removal S-Traps					
Moisture S-trap, preconditioned*				5060-9084	
Moisture S-trap, unconditioned				5060-9077	
Big Moisture Traps (BMT Series)					
Big Moisture Trap	750			BMT-2	BMT-4
Refill for Big Moisture Trap (enough for 2 refills)				BMSR-1	BMSR-1

**Traps can be reconditioned by heating at a minimum of 350°C, with flow for 6 hours*

Refillable glass moisture trap

Moisture S-trap

Big moisture trap

Hydrocarbon Traps

Hydrocarbon traps remove organics, such as hydrocarbons and halocarbons, from the gas stream. The adsorbent is usually activated carbon or an impregnated carbon filter media. Carbon removes organic solvents from the gas stream, including the typical solvents used in nearly every lab. Hydrocarbon-moisture combination traps are also available which remove water in addition to organics. Capillary grade hydrocarbon traps are purged with ultra-high helium and packed with a very efficient activated carbon material. Metal trap bodies are used to prevent any contaminants in plastic trap bodies from contaminating the carbon adsorbent. Most hydrocarbon traps can be refilled by the end user.

Hydrocarbon trap, HT200-2

Hydrocarbon Traps

Description	Size (cc)	1/8 in. Part No.	1/4 in. Part No.
Hydrocarbon Traps (HT Series)			
Hydrocarbon Trap	200	HT200-2	HT200-4
Adsorbent Refill (1 pint)		ACR	ACR
Big Hydrocarbon Traps (BHT Series)			
Big Hydrocarbon Trap	750	BHT-2	BHT-4
Refill for Big Hydrocarbon Trap (enough for two refills)		BACR	BACR
Hydrocarbon Removal S-Traps			
Hydrocarbon S-Trap, used for trapping organics from gases		5060-9096	
Capillary Grade Hydrocarbon Traps (HT3 Series)			
Capillary Grade Hydrocarbon Trap	100	HT3-2	HT3-4
Adsorbent Refill (1 pint)		ACR	ACR

Tips & Tools

Hydrocarbon traps should be used with carrier, FID and ELCD gases.

Oxygen Traps

Oxygen traps usually include a metal-containing inert support reagent. Most oxygen traps reduce the oxygen concentration to below 15-20 ppb. The capacity of a standard oxygen trap is approximately 30 mg of oxygen per 100 cc of trap volume. Oxygen traps can also remove some small organic and sulfur compounds from gas streams, but this is not their primary application.

Indicating oxygen trap, IOT-2-HP

Metal (usually aluminum) trap bodies are recommended for GC analyses. Some plastics are permeable to air and contain contaminants that can degrade gas quality. In addition, many of the metal bodied oxygen traps can withstand high pressures (up to 2000 psi). Some oxygen traps also remove moisture from the gas stream without affecting the oxygen removal capability.

Indicating oxygen traps change color when oxygen is present in the gas at harmful levels. Indicating traps are not intended to be the primary oxygen removal trap, but should be used in conjunction with a high capacity non-indicating oxygen trap. They are installed after the high capacity oxygen trap in the gas line to indicate when the high capacity trap has expired and needs to be changed. Expired oxygen traps need to be immediately changed since they can contaminate the gas, in addition to failing to remove oxygen.

Tips & Tools

Oxygen traps are critical on carrier gas supplies to prevent column bleed. They are also recommended with ECD.

Oxygen Traps

Description	Size (cc)	1/8 in. Part No.	1/4 in. Part No.
Indicating Oxygen Traps (IOT and LIOT Series)			
Indicating Oxygen Trap	30	IOT-2-HP	IOT-4-HP
Large Indicating Oxygen Trap	150	LIOT-2	LIOT-4
Economy Non-Indicating Oxygen Traps (OT1 Series)			
Oxygen Trap	70	OT1-2	OT1-4
Big Oxygen Traps (BOT Series)			
Big Oxygen Trap	750	BOT-2	BOT-4

Combination Traps

Agilent carries several Combination Traps that provide multiple contaminant removal in a single trap. These traps offer:

- Optimized adsorbents for maximum surface area and capacity.
- Leak-free, one-piece design to eliminate potential leaks from using multiple traps.
- Efficient design which prevents channeling and promotes efficient scrubbing.
- The ultimate in purification with the Big Universal Trap, which removes oxygen, moisture, hydrocarbons, carbon monoxide, and carbon dioxide.

Agilent OT3 trap

Combination Traps

Description	Size (cc)	1/8 in. Part No.	1/4 in. Part No.
Oxygen/Moisture Traps (OT3 Series)			
OT3 Trap	100	OT3-2	OT3-4
Hydrocarbon/Moisture Traps (HMT Series)			
Hydrocarbon/Moisture Trap	200	HMT200-2	HMT200-4
Adsorbant Refill (1 pint)		HCRMS	HCRMS
Combination Traps for Chemical Ionization MS			
Chemical Ionization for MS*		G1999-80410	
Big Universal Traps (RMS Series)			
Hydrogen		RMSHY-2	RMSHY-4
Helium (Ar/Me)		RMSH-2	RMSH-4
Nitrogen		RMSN-2	RMSN-4

**Isobutane or methane applications only*

NEW! Renewable Gas Purification System

The Renewable Gas Purification System from Agilent not only traps large quantities of contaminants and lasts a long time, but it is also recyclable. With average use, you'll only have to purchase a replacement cartridge once per year or after approximately 20 cylinders worth of purification. And when you need a replacement, you have the option to purchase a new or recycled cartridge. Recycled cartridges are refilled and certified to the full specification of the new cartridges.

- All cartridges are environmentally friendly and reduce waste.
- High capacity – 850cc or more Oxygen filtration, 12g H_2O, and approximately 8g hydrocarbon filtration per cartridge – in a compact footprint.
- Improves 99.995% gas purity to 99.9995% purity.
- Dual indicators make it easy to see the purification results.
- Labeling displays indicator color and shape for accurate reading.
- Simple twist on/off knob and guide rod make cartridge changes quick and easy.
- One Renewable Purifier system can support up to four GC systems.
- Designed with efficiency, safety, and environmental responsibility in mind.

Renewable gas purification system

Renewable Gas Purification System

Description	Part No.
Renewable gas purifier system startup kit Includes one G3440-80007 base, one G3440-60003 renewable gas purifier cartridge, and wall or bench mount hardware.	G3440-60004
Renewable gas purifier system, base only	G3440-80007
Renewable gas purifier cartridge, new	G3440-60003
Renewable gas purifier recycled cartridge	G3440-69003

Split vent trap and cartridges, RDT-1020

Universal/External Split Vent Trap

Split vent traps stop environmental pollution. The split vent trap was designed to protect the lab environment from the contaminants released by split injection systems, which can vent up to 500 times the amount of sample reaching the detector into the laboratory's air. A replaceable, impregnated carbon filter media traps and eliminates a broad range of contaminants. The traps are also easy to change and come with three packs of replacement cartridges each. Replace approximately every six months.

Universal/External Split Vent Trap

Description	Part No.
Universal/external split vent trap with 3 cartridges, 1/8 in. Swagelock fitting	RDT-1020
Replacement cartridges, 3/pk	RDT-1023
Split vent trap kit	G1544-60610

Tips & Tools

Replace split vent traps every six months.

Regulators

Agilent recommends using our economical brass body, dual stainless steel diaphragm regulators for most GC applications. These regulators, combined with the proper gas purification system, provide proper gas pressure control and purity for gas chromatography.

Brass body regulator

Brass Body, Dual Stainless Steel Diaphragms, 1/8 in., U.S. Only*

Description	Part No.
CGA 346, 125 psig max (8.6 bar), Air	5183-4641
CGA 350, 125 psig max (8.6 bar), H_2, Ar/Me	5183-4642
CGA 540, 125 psig max (8.6 bar), O_2	5183-4643
CGA 580, 125 psig max (8.6 bar), He, Ar, N_2	5183-4644
CGA 590, 125 psig max (8.6 bar), Air	5183-4645

**For 1/4 in. tubing, purchase a 1/4 in. adapter, U.S. only*

Connectors (Swagelok to Female NPT)

Description	Unit	Part No.
1/8 in. (A) x 1/4 in. (B) connector, brass (included with brass regulators)	1/ea	0100-0118
1/4 in. (A) x 1/4 in. (B) connector, brass*	1/ea	0100-0119

**Required for plumbing 1/4 in. tubing to regulators*

Tips & Tools

When ordering a regulator, be sure to specify the proper connections. In the US, most gas manufacturers follow CGA connection guidelines. In Europe, there are a number of organizations designating cylinder connections that are specific to individual countries. It is best to contact your local supplier for the proper connection designation.

Tubing

Cleaning Tubing

Before any tubing is placed into service, or if it becomes contaminated with use, it is essential that it be properly cleaned. Unclean or improperly cleaned tubing can lead to contamination of the system with disastrous results. Never use chlorinated solvents to clean tubing or fittings.

Agilent provides clean, high quality GC grade tubing for large systems as an economical alternative.

Agilent recommends using copper tubing for most applications, since it is easy to bend and plumb and is less expensive than stainless steel. Use stainless steel tubing only for crucial applications that require very high purity, or where building codes mandate its use.

Determining Tubing Length

Parameters: 2000 sccm (4.2 scfh); Temperature: 70°F; Pressure 30 psig

Tubing Type	Diameter (inches)	Recommended Max. Length (feet)	Pressure Drop (psig)
Copper	1/8	50	2
Copper	1/4*	300	0.5

**Recommended when multiple instruments are connected to the same source*

Tubing - Precleaned

Description	Part No.
Copper tubing, 1/8 in., 50 ft	5180-4196
Copper tubing, 1/8 in., 12 ft	5021-7107

Fittings

Description	Part No.
Fittings Kit Includes brass nuts, ferrules, caps, plugs, unions and reducers	5180-4161
1/8 in. brass nut and ferrule set	5080-8750

Cylinder wall bracket, 5183-1941

Cylinder wall bracket

Description	Part No.
Cylinder wall bracket with strap & chain (cylinder size up to 14 in., 35 cm)	5183-1941

Flowmeters

Setting and maintaining GC flow rates greatly affects the instrument accuracy and sensitivity. During maintenance, verify carrier and support gas flows with the proper flowmeter.

Precision gas flow meter, 5067-0223

NEW! Precision Gas Flow Meter

Agilent's Precision Gas Flow Meter is the ultimate gas flowmeter for chromatography applications. This hand-held flowmeter incorporates industry leading performance and features in a highly accurate and reliable package. The inherent stability of the rugged, solid state components allows us to provide the longest calibration interval on the market, all traceable to NIST standards.

- Highly accurate and reliable measurement of common carrier and fuel gases used in GC, including nitrogen, air, carbon dioxide, hydrogen, helium, and argon/methane.
- Two year guaranteed calibration period traceable to NIST standards.
- Measures flow (0-500 mL/min.) based on gas viscosity properties with an accuracy of ± 0.8% of reading + 0.2% of full scale.
- Flow rate range from 0-500 mL/min (no conversion necessary from SCCM) eliminates the need for two separate meters to measure capillary and standard flows.
- Displays mass flow, volumetric flow, temperature, and pressure readings simultaneously.
- Can be plumbed inline.

Precision Gas Flow Meter

Description	Part No.
Precision gas flow meter	5067-0223

ADM flowmeters

ADM 1000

- Accuracy ± 3%.
- Operating temperature range – 0 to 45ºC for the instrument, -70 to 135ºC for the tubing.
- Calibration – traceable to NIST primary standards.
- Real time, split ratio measurement.
- CE mark certified.
- Measures flow rates from 0.5 to 1000 mL/min.
- Split ratios – compare the ratio from one gas measurement to another (i.e., injection port split ratios).

ADM 2000

In addition to the features of the ADM 1000, the ADM 2000 includes:

- Mass flow measurements – measure flow rate, independent of atmospheric pressure and temperature (calculated).
- Data output through RS-232 port.
- 9V battery and AC power adapter (120 or 220 VAC).

ADM Flowmeters

Description	Flow Rate (mL/min)		Gases Measured*	Accuracy (%)	Power Supply	RS-232 Data Output	Part No.
	Low	High					
ADM 1000**	0.5	1000	All	± 3	9V Battery	None	220-1170
ADM 2000**	0.5	1000	All	± 3	Battery or 120V AC	Yes	220-1171-U
ADM 2000E**	0.5	1000	All	± 3	AC Adapter, 220V	Yes	220-1171-E
Carrying Case for ADM							907-0056

**± 3% or ± 0.2 mL/min, whichever is greater with a flow rate of 0.5 - 1,000 mL/min*

***Non-corrosive gases only*

NEW! Gas Leak Detector

Gas leaks can cause detector noise and baseline instability, shorten column life and waste expensive carrier gas. Liquid leak detectors, although inexpensive, can contaminate your system. Agilent's G3388A electronic leak detector is an easy way to quickly identify leaks in your system.

Gas leak detector

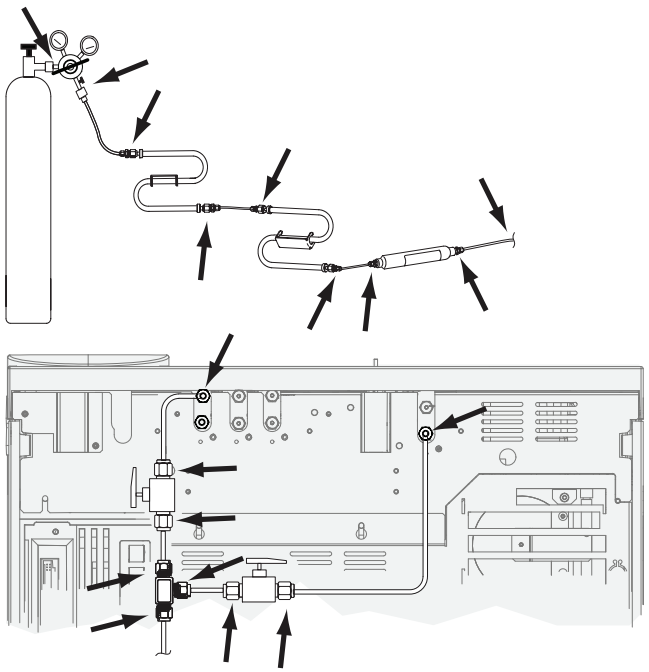
Gas Leak Detector

Description	Part No.
Handheld electronic gas leak detector Includes probe, unit, AC power adapter/battery charger, battery, and user manual.	G3388A

Check valves, fittings, and traps for leaks after every maintenance and thermal cycling, as these can loosen some types of fittings.

Check for leaks at these connections:

- Gas supply bulkhead fittings
- Gas cylinder fitting
- Regulator fittings
- Traps
- Shut-off valves
- T-fittings



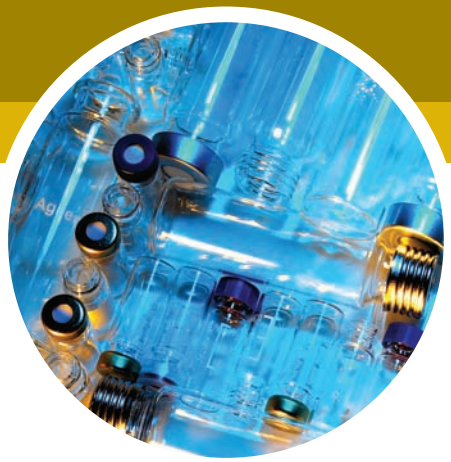
Sample Introduction

Ensure optimal peak shape and reproducibility through accurate sample delivery.

They may be small, but sample introduction supplies can have a profound impact on your results. That is why Agilent vials, septa, and syringes are meticulously engineered to work seamlessly with your GC and GC/MS instruments. They can help you achieve reliable, repeatable results even for your most complex runs.

This section guides you through...

- The selection, use, and maintenance of your essential sample introduction supplies.
- Troubleshooting common and unusual syringe problems.
- Essential procedures, such as vial filling, syringe cleaning, and choosing the right septa material for your application.



Vials

Agilent's wide opening vials are designed specifically for analyzing samples with your GC. They have specially designed vial neck angles, bottom design, and height to ensure compatibility with Agilent autosamplers with rotating or robotic arm trays. Agilent offers a large variety of autosampler vials in different closures, cap colors, septa choices, and package options. Agilent also offers convenience packs with 500 vials and caps in a reusable blue storage box.

For small samples sizes, Agilent offers a variety of options. You can use microvolume inserts with the wide opening vials or, for added convenience, use vials with small volume capacity.

Agilent Certified Vials

Agilent certified vials undergo stringent testing of critical dimensions to ensure that each vial works flawlessly with your GC and GC/MS systems. Additionally, each certified vial is made from first hydrolytical class, borosilicate glass type 1 then packed in a clean, sealed environment to prevent contamination.

Vial Options

Glass - for general purpose use and for use with acids

Silanized - for use with samples that bind to glass, and for trace analysis

Polypropylene - for use with alcohols and aqueous solvents

High Recovery Vials - for use with limited sample vials

Amber Vials - for use with light-sensitive samples

Microvolume Inserts - for use with very small sample volumes

Vial Filling

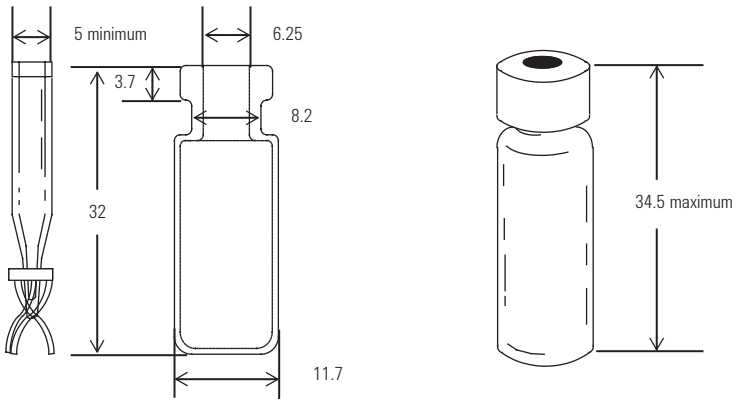
When filling sample vials, keep in mind:

- If you need to test a large amount of sample over repeated injections, divide the sample among several vials to obtain reliable results.
- When sample volume in the vial is low, contaminants from the previous sample injection or solvent washes may have a greater impact on the sample.

The airspace in the vial is necessary to avoid forming a vacuum when sample is withdrawn. This could affect reproducibility.

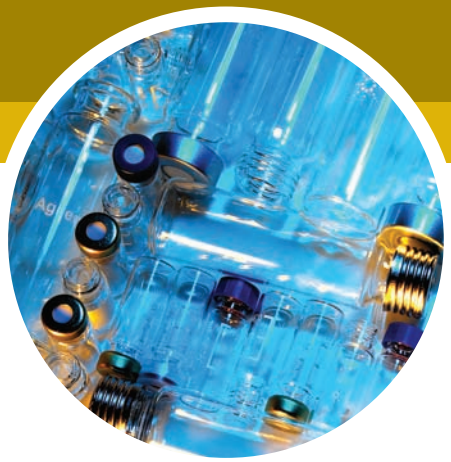
Preparing Sample Vials

The Agilent 7683 Automatic Liquid Sampler Injector and the tray use clear or amber glass sample vials with crimp, snap, or screw top vials. The following diagram shows the critical dimensions for sample vials and microvial inserts to be used with the 7683 Automatic Liquid Sampler. These dimensions do not make up a complete set of specifications. Incompatible sample vials cause tray and turret errors.



Microvial insert Crimp cap sample vial Maximum height of a capped vial

All dimensions in millimeters



Selecting Vial Septa Materials

Vial cap septa are critically important to optimal analysis. Each septum complements the overall system and enhances chemical performance. Agilent's vial cap septa are specifically formulated and constructed for optimum system performance, with minimal coring and superior chemical inertness.

Type	Uses
Red Rubber/PTFE	• Routine analysis • Moderate resealing • Excellent chemical inertness • Not recommended for multiple injections or storage of samples • Least expensive
Silicone/PTFE	• Excellent resealing • Resists coring • Good for multiple injections
PTFE/Silicone/PTFE	• Used in trace analysis applications • Above average resealing • Most resistant to coring • Least evaporation • Use with large diameter, blunt tip syringe needles
PTFE Disc	• Good for MS and ECD analysis • Good for large-volume injections • Chemically inert • No resealing • Single injection • No long-term sampling storage
Viton	• Chlorinated solvents • Organic acids • Limited resealing • Not suitable for 32 guage syringe

Tips & Tools

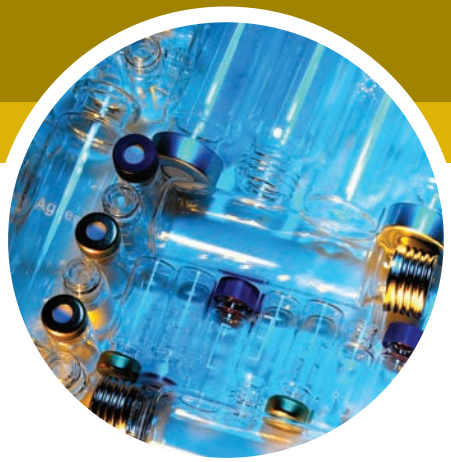
To determine potential septa quantities, consider:

- The number of samples run during a day/week.
- If samples are run in small or large batches.
- If samples are run manually or with an autosampler.
- If samples are run overnight, unattended.

Convenience vial and cap pack

Convenience Vial and Cap Packs

Vials	Caps	Unit	Part No.
Certified Screw Top Vial Convenience Packs			
Clear	Blue screw caps, PTFE/red rubber septa	500/pk	5182-0732
Clear with write-on spot	Blue screw caps, PTFE/red rubber septa	500/pk	5182-0867
Amber with write-on spot	Green screw caps, PTFE/red rubber septa	500/pk	5182-0733
Clear	Blue screw caps, PTFE/silicone/PTFE septa	500/pk	5182-0736
Clear with write-on spot	Blue screw caps, PTFE/silicone/PTFE septa	500/pk	5182-0869
Amber with write-on spot	Green screw caps, PTFE/silicone/PTFE septa	500/pk	5182-0737
Clear	Blue screw caps, PTFE/silicone septa	500/pk	5182-0734
Clear with write-on spot	Blue screw caps, PTFE/silicone septa	500/pk	5182-0868
Amber with write-on spot	Green screw caps, PTFE/silicone septa	500/pk	5182-0735
Clear	Blue screw caps, pre-slit PTFE/silicone septa	500/pk	5183-2079
Clear with write-on spot	Blue screw caps, pre-slit PTFE/silicone septa	500/pk	5183-2080
Amber with write-on spot	Green screw caps, pre-slit PTFE/silicone septa	500/pk	5183-2081
Clear	Blue screw caps, pre-slit PTFE/silicone septa	500/pk	5067-0205



Convenience Vial and Cap Packs

Certified Crimp Top Vial Convenience Packs			
Clear	Silver aluminum crimp caps, PTFE/red rubber septa	500/pk	5181-3400
Amber with write-on spot	Silver aluminum crimp caps, PTFE/red rubber septa	500/pk	5181-8801
Snap Top Vial Convenience Packs			
Clear	Clear polypropylene snap caps, PTFE/red rubber septa	500/pk	5182-0547
Amber, with write-on spot	Clear polypropylene snap caps, PTFE/red rubber septa	500/pk	5182-0548

Certified crimp top vial convenience pack

Electronic crimper, 5062-0207

Electronic Crimpers and Decappers

Whenever large amounts of crimp vials need to be crimped or decapped, the electronic crimper or decapper is the right tool. It reduces stress and repetitive motion injury associated with manual plier-style crimpers and decappers. Agilent's newly-designed crimper offers easy, hand-held pushbutton operation and provides the following advantages:

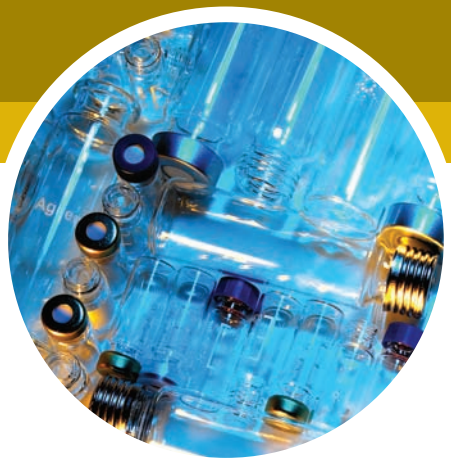
- Stronger and sturdier crimping and decapping.
- Consistent seals.
- Shorter recharging time and a larger number of battery charges.
- Better clearance and more flexibility thanks to improved crimp jaws.
- Individual test certificates.

Electronic Crimpers and Decappers

Description	Part No.
11 mm Electronic Crimper with 4.8v rechargeable battery pack and charger	5062-0207
20 mm Electronic Crimper with 4.8v rechargeable battery pack and charger	5062-0208
11 mm Electronic Decapper with 4.8v rechargeable battery pack and charger	5062-0209
20 mm Electronic Decapper with 4.8v rechargeable battery pack and charger	5062-0210
4.8v nickel metal hydride replacement battery	5188-6565

Tips & Tools

For a consistent seal, make sure there are no folds or wrinkles on the part of the cap that wraps under the neck of the vial.



Gold Standard Autosampler Syringes

With a broad selection of syringes for auto injection, Agilent has what you need for accurate and effective sampling. Agilent delivers even more value in every autosampler syringe with the introduction of many new features in our line of Gold Standard GC Autosampler Syringes.

Gold standard autosampler syringes

Agilent Gold Standard Autosampler syringes are designed:

- For reproducible sample volume delivery.
- Specifically for the Agilent inlet or autosampler.
- To maximize inlet septum lifetime.

Agilent Gold Standard Autosampler syringes feature:

- Lot numbers printed directly on the barrel with a corresponding Certificate of Conformance ensuring certified performance to all specifications.
- Gold protective cap on the fused needle, preventing the glass syringe barrel from chipping as it is pressed against the inlet.
- Black ink and gold illuminating backing strip, for effortless viewing of the volume scale, which is easily discernible from imitators.
- Environmentally friendly packaging, an improved design that reduces waste.
- Individually sealed packaging, for contaminant-free use right out of the box.

Tips & Tools

Use syringe needles with an Agilent dual-taper needle or a conical tip. Sharp-tipped needles tend to tear the inlet septum and cause leaks. Also, a sharp-tipped needle tends to leave residual amounts of sample on the septum as it exits, resulting in a large solvent tail on the chromatogram.

Needle Gauge Selection

- Syringe needles compatible with Agilent Automatic Liquid Samplers are 42 mm long and have an HP style or cone shaped tip.
- The Merlin Microseal requires 23 gauge needles.
- The smaller the gauge, the larger the needle diameter.

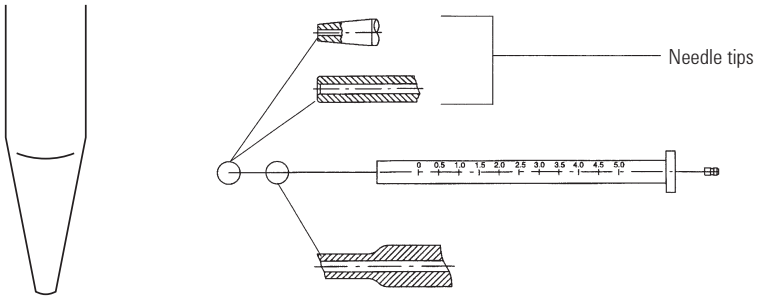
Needle Gauge Selection

Inlet	Needle Gauge	Column Type
Packed, split or splitless (including PTV)	23 gauge or 23/26 gauge tapered	Any
Cool on-column	23/26 gauge tapered or 26 gauge	530 µm
Cool on-column	26/32 gauge tapered	320 µm
Cool on-column	26/32 gauge tapered	250 µm

Tips & Tools

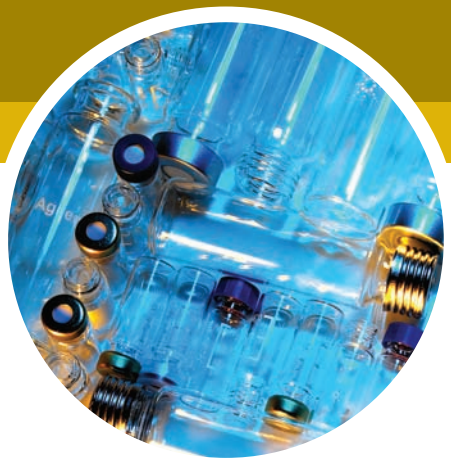
For highest productivity and to minimize coring, use Agilent Premium Non-Stick Inlet Septa with Center Point Guide.
See page 46.

Needle Tip **Needle Shape**



Cone tip

Tapered needle



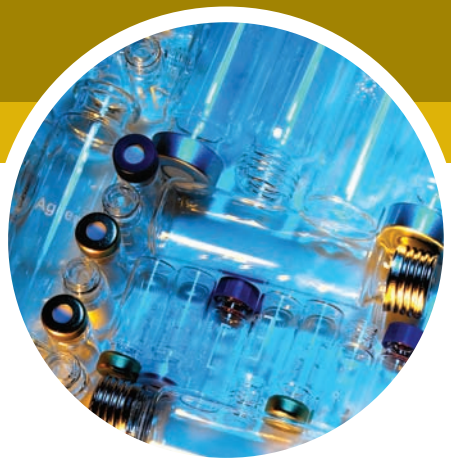
Syringe Characteristics and Recommended Uses

Syringe	Advantage	Limitations	Recommended Use
10 µL, PTFE-tipped	• Less plunger binding than fitted plunger • Replaceable plunger for reduced repair cost • Tight seal between plunger and barrel	• More expensive than fitted plunger • PTFE-tipped syringes not available in 5 µL size	• High sample throughput • Samples in polar solvents • Dirty samples • Gases and volatile samples • Reactive samples
10 µL, fitted plunger	• Most economical • Most reliable fitted plunger syringe • Less bending • Better for high viscosity samples	• Most accurate only for 1 µL and larger injections • Plunger not replaceable	• General purpose syringe • Clean samples • Routine analysis
5 µL, fitted plunger	• Most accurate for 1 µL injection • No hardware modification needed for 0.5 µL	• Thinnest plunger, can bend more easily • Not ideal for higher viscosity samples • Plunger not replaceable	• 1 µL injections • Clean samples • Routine analysis

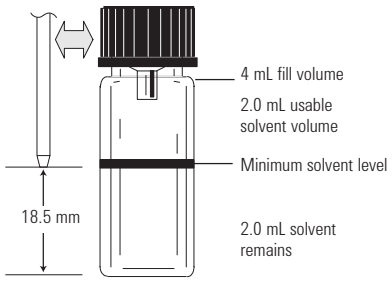
Syringe Troubleshooting Guide

Proper care, cleaning, and handling of each syringe will help ensure correct performance and long life. When cleaning your syringe, it is best to use solvents that effectively dissolve the sample you are working with. Try to avoid cleaning agents that are alkaline, contain phosphates, or are strongly acidic.

Problem	Possible Cause(s)	Suggested Action(s)
Bent plungers or stuck syringes	• Particles such as dust, leftover samples, salts, metal, or glass can fill the narrow gap between the plunger shaft and the inside wall of the barrel.	• Try PTFE-tipped plunger syringe. • If the plunger's movement feels "gritty," remove the plunger from the barrel, flush the shaft with solvent, and wipe it dry with a lint-free cloth. Then, carefully insert the plunger back into the barrel. Finally, submerge the tip of the needle into a container of solvent, and cycle the plunger to pull the solvent into and out of the barrel. • Never cycle the plunger in a dry syringe. • Do not "mix & match" plungers and barrels. • Always clean syringes after use immediately.
Bent needles	• Improper needle alignment. • Narrow-gauge needles (26 gauge) bend more easily than larger (23 gauge) needles. • Needles tend to bend when inserted into the sample vial - not the inlet port. This can be caused by septa that are too "tough." • If the needle has been slightly bent when mounted in the autosampler - or when the syringe is installed into the autosampler - then it is more likely to bend further when it pushes through the septa on the sample vial caps.	• Use only Agilent autosamplers. They are precision-designed to ensure proper alignment with the Agilent inlet. • Use 23 to 26-gauge tapered needles to get the combined benefits of greater septa life and fewer bent needles. • Only use Agilent vials and septa.



Problem	Possible Cause(s)	Suggested Action(s)
Blocked needles	• Sample material or contaminants may be trapped inside the needle. • The needle may not have been properly cleaned.	Remove the plunger and use a second syringe to fill the blocked syringe with solvent. Then, insert the plunger and gently push solvent through needle. Important: Try to use a cleaning agent that is appropriate for the contaminant. Common choices are methanol, methylene chloride, acetontrile, and acetone.
Rust Note: even minor rust can cause the plunger to become stuck in the barrel.	• During normal use, the shaft rubs against the glass walls of the barrel. This gradually wears away the rust-resistant metal on the shaft's surface. • Rusting happens most rapidly when using water or solvents that may contain (or absorb) water.	To slow this process, remove the water from the syringe at the end of each day. 1. Rinse the syringe several times with a "dry" solvent, such as acetone. 2. Remove the syringe from the autosampler, and wipe the plunger dry with a lint-free cloth. 3. Let syringe and plunger air dry.
"Ring around the neck" (A dark ring between the top of the barrel and the end of the volume scale.)	• Skin oils and other organic material. • Fine metal and glass particles from the syringe plunger and barrel may be rubbing together. Once this happens, the plunger may bend if used further.	• Never touch the plunger shaft with your fingers. • If build-up appears when water is the solvent: rinse syringe with acetone and wipe the plunger clean at the end of each day.
Loose plungers Accompanied by syringe leaks and area count reproducibility problems.	• The syringe is nearing the end of its useful life.	• Replace the syringe. • Note: Plungers normally feel "loose" when non-polar solvents (like hexane and toluene) are used.



Needle tip position when withdrawing solvent

Cleaning Syringes During an Autosampler Sequence

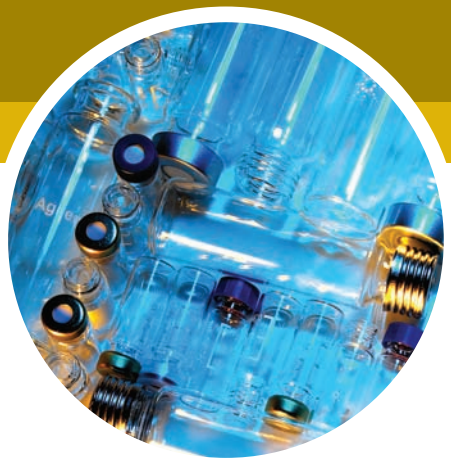
To ensure the syringe is properly cleaned between injections, rinse and fill each solvent bottle with 4 mL of fresh solvent. The liquid level will be near the shoulder of the bottle. Good laboratory practice dictates using no more than 2 mL of the 4 mL solvent for syringe washes. The needle tip draws solvent 18.5 mm from the bottom of the vial.

Tapered Needle, 23-26s Gauge Autosampler Syringes

Volume (µL)	Description	Unit	Needle	Part No.
5	Tapered, fixed		23-26s/42/HP	5181-1273
	Tapered, fixed	6/pk	23-26s/42/HP	5181-8810
	Tapered, removable		23-26s/42/HP	5182-0835
	Replacement needle for 5 µl syringe	3/pk	23-26s/42/HP	5182-0832
10	Tapered, fixed		23-26s/42/HP	5181-1267
	Tapered, fixed	6/pk	23-26s/42/HP	5181-3360
	Tapered, removable		23-26s/42/HP	5181-3321
	Replacement needle for 10 µl syringe	3/pk	23-26s/42/HP	5181-3319
	Tapered, fixed, PTFE-tipped plunger		23-26s/42/HP	5181-3354
	Tapered, fixed, PTFE-tipped plunger	6/pk	23-26s/42/HP	5181-3361
	Replacement plunger with PTFE tip for fixed needle 10 µl syringe			5181-3365
	Tapered, removable		23-26s/42/HP	5181-3356
	Replacement plunger with PTFE tip for removable needle 10 µl syringe			5181-3358
50	Tapered, fixed, PTFE-tipped plunger		23-26s/42/HP	5183-0314
100	Tapered, fixed, PTFE-tipped plunger		23-26s/42/HP	5183-2042

Tips & Tools

When cleaning syringes between injections, use Agilent wash vials (p/n 9301-0723) and diffusion caps (p/n 07673-40180).

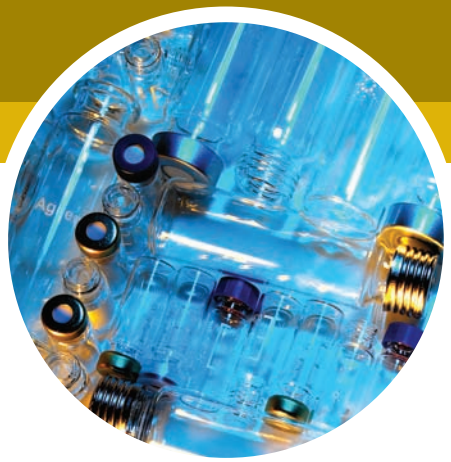


Straight Needle, 23 and 26s Gauge Autosampler Syringes

Volume (µL)	Description	Unit	Needle	Part No.
0.5	Cone-tipped, 23 gauge		23/42/HP	5188-5246
1	Cone-tipped, 23 gauge		23/42/HP	5188-5247
5	Straight, fixed, 26 gauge		26s/42/HP	9301-0891
	Straight, fixed, 26 gauge	6/pk	26s/42/HP	5183-4728
	Straight, fixed, 23 gauge		23/42/HP	9301-0892
	Straight, fixed, 23 gauge	6/pk	23/42/HP	5182-0875
	Straight, removable, 23 gauge		23/42/HP	5182-0834
	Replacement needle for 5 µl syringe	3/pk	23/42/HP	5182-0830
10	Straight, fixed, 26 gauge		26s/42/HP	9301-0714
	Straight, fixed, 26 gauge	6/pk	26s/42/HP	5183-4729
	Straight, fixed, 23 gauge		23/42/HP	9301-0713
	Straight, fixed, 23 gauge	6/pk	23/42/HP	9301-0725
	Straight, fixed, PTFE-tipped plunger		23/42/HP	5181-8809
	Straight, fixed, PTFE-tipped plunger	6/pk	23/42/HP	5183-4730
	Replacement plunger for 10 µl fixed needle syringe			5181-8808
	Straight, removable, 23 gauge		23/42/HP	5181-8806
	Straight, removable, PTFE-tipped plunger		23/42/HP	5181-8813
	Replacement needle for 10 µl syringe	3/pk	23/42/HP	5181-8811
	Replacement plunger with PTFE tip for removable needle 10 µl syringe			5181-3358
25	Straight, fixed, PTFE-tipped plunger		23/42/HP	5183-0316
50	Straight, fixed, PTFE-tipped plunger		23/42/HP	5183-0318
100	Straight, fixed, PTFE-tipped plunger		23/42/HP	5183-2058

GC Automatic Liquid Sampler Supplies

Description	Unit	Part No.
4 mL Clear screw top wash vials with screw caps (no septa)	144/pk	9301-0723
Septa for 4 mL vial	144/pk	9301-1031
Diffusion inserts for 4 mL vials	12/pk	07673-40180
4 mL wash vials with fill markings and caps	25/pk	5182-0551
Screw for mounting syringe		07673-20570
Quadrant tray (4 tray sections)		18596-40015
7673 Basic Supply Kit Contains 10 µL syringes (6/ea), 23/26 gauge needles, 4 mL vials with diffusion caps (144/pk), 2 mL automatic sampler vials with screw caps (1,000/pk), GC septa (25/pk), vial racks (5/pk)		07673-60840



Headspace Sampler

The appropriate maintenance frequency for the Headspace Sampler varies significantly depending on the sample matrix, solvents, temperatures, and sample throughput.

G1888 Headspace sampler

Recommended Maintenance Schedule

Maintenance Activity	Low Boiling Solvents (water, food, flavor analyses)		High Boiling Solvents (OVI, blood alcohol analysis)	
	≤ 70 samples/day	> 70 samples/day	≤ 70 samples/day	> 70 samples/day
Replace sample probe	6 months	3 months	3 months	Every month
Steam clean	6 months	3 months	3 months	Every month
Replace loop and deactivated tubing	With PM	With PM	With PM	6 months
Check tray tension and alignment	12 months	6 months	12 months	6 months
PM for headspace and inlet	36 months	12 months	12 months	6 months
Extended PM for headspace	60 months	36 months	24 months	12 months

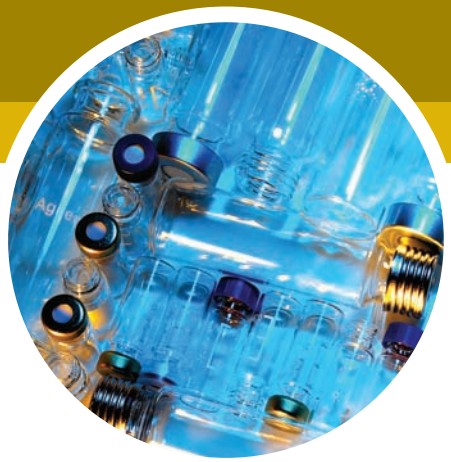
Certified headspace vials

Headspace Vials

Description	Unit	Part No.
Certified Flat Bottom Headspace Crimp Top Glass Vials		
20 mL, clear, 23 x 75 mm	100/pk	5182-0837
10 mL, clear, 23 x 46 mm	100/pk	5182-0838
Certified Rounded Bottom Headspace Crimp Top Glass Vials		
20 mL, clear, 23 x 75 mm	100/pk	5183-4474
10 mL, clear, 23 x 46 mm	100/pk	5183-4475
Certified Headspace Vials for G1888A Autosampler		
Headspace screw top vial, 20 mL, clear, 23 x 75 mm	100/pk	5188-2753
Headspace screw top vial, 10 mL, clear, 23 x 46 mm	100/pk	5188-5392
Headspace screw top vial, 20 mL, amber, 23 x 75 mm	100/pk	5188-6537
Headspace screw top vial, 10 mL, amber, 23 x 46 mm	100/pk	5188-6538
Certified UltraClean 18 mm screw caps with septa for headspace vials	100/pk	5188-2759

Headspace Vial Convenience Kits

Description	Specifications	Unit	Part No.
20 mL Headspace crimp top, flat bottom vials, silver aluminum one-piece crimp caps with safety feature, molded gray PTFE/black butyl septa	< 125°C	100/pk	5182-0839
20 mL Headspace crimp top, flat bottom vials, silver aluminum one-piece crimp caps with safety feature, PTFE/white silicone septa	< 180°C	100/pk	5182-0840



Headspace supplies

Headspace Supplies

Description	Part No.
Stainless Steel Sample Loops	
Sample loop, 1 mL, deactivated	2321700003
Sample loop, 3 mL, deactivated	2321700004
Probes and Unions	
Sample probe, deactivated	2322700011
M6 union, brass	2302533140
Union, zero dead volume, deactivated	2307230001
Union	2307232901
Transfer Line Needles and Unions	
Needle, headspace transfer line, deactivated 0.5 mm OD	2322590004
Needle, headspace transfer line, deactivated 0.7 mm OD	2322590005
Strain relief septum nut	6410090050
Tubing	
Tubing, solenoids to 6-port, deactivated	0410105017
Tube, probe to 6-port valve, deactivated	1300502506
Standards	
OQ/PV Headspace Sample Contains 0.2-0.3% t-butyl disulfide, 1,2-dichlorobenzene, and nitrobenzene in ethanol	5182-9733
PM Kits	
G1888A PM kit with 1 mL loop	G1888-60702
G1888A PM kit with 3 mL loop	G1888-60703
G1888A enhanced PM kit	G1888-60704

CTC Autosampler Supplies

Agilent now offers a portfolio of CTC-recommended supplies for your GC PAL and CombiPAL autosamplers.

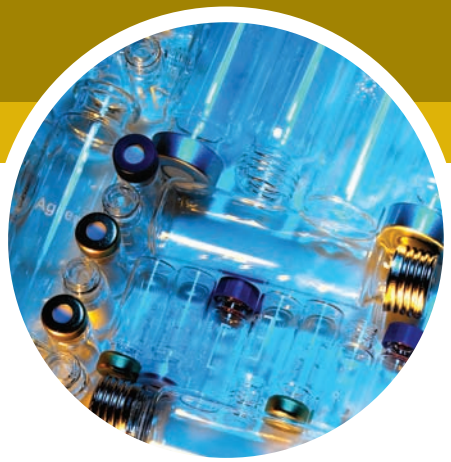
CombiPAL and GC PAL Liquid Injection Vials and Caps

These micro and 2 mL vials and caps are designed to work seamlessly with CombiPAL and GC PAL magnetic needle guides and bar code readers.

CombiPAL Autosampler

CombiPAL and GC PAL Liquid Injection Vials and Caps

Description	Unit	Part No.
2 mL vials		
Crimp top vial, wide opening, clear	100/pk	5181-3375
Crimp top vial, wide opening, amber, write-on spot	100/pk	5181-3376
Crimp top vial, wide opening, clear, write-on spot	1000/cs	5183-4492
Crimp top vial, wide opening, amber, write-on spot	1000/cs	5183-4493
Screw top vial, wide opening, clear	100/pk	5182-0714
Crimp/snap top vial, wide opening	100/pk	5182-0544
2 mL caps		
Crimp cap, 11 mm magnetic	100/pk	5188-5386
Screw cap, PTFE/white silicone septa	100/pk	5182-0720
Snap cap, blue polyethylene, PTFE/silicone septa	100/pk	5182-0541
Micro vials		
Crimp top vial, 0.8 mL, amber glass, flat bottom	1000/pk	5183-4487
Crimp top vial, 0.1 mL, clear, tapered	500/pk	5180-0844
Crimp top vial, 0.3 mL, clear, round	500/pk	5180-0841
Crimp top vial, 0.7 mL, amber, round	500/pk	5180-0805
Crimp top vial, 0.5 mL, amber, conical	500/pk	5180-0806
Micro caps		
Crimp caps with PTFE/silicone septa	500/pk	5180-0842



CombiPAL and GC PAL Liquid Injection Syringes

A key feature of CTC's GC PAL and CombiPAL systems is the ability to inject a wide range of sample volumes – up to 500 µL for LVI applications. To help you take full advantage of this flexibility, Agilent offers a wide range of C-type syringes – from 1.2 µL through 500 µL – to accommodate fast and large-volume injections. Each syringe is subjected to stringent quality control procedures to make sure it meets the highest levels of precision and accuracy.

CombiPAL and GC PAL Liquid Injection Syringes

Volume (µL)	Description	Unit	Gauge	Needle	Part No.
1.2	Fixed needle		26	26/51/AS	G6500-80113
5	Fixed needle		26	26/51/AS	G6500-80114
10	Fixed needle		26	26/51/2	G6500-80115
			26	26/51/AS	G6500-80116
	Replacement plunger	10/pk			G4200-80105
	Fixed needle		23	23S/51/AS	5188-6596
25	Fixed needle		26	26/51/AS	G6500-80117
	Replacement plunger	10/pk			G4200-80104
100	Fixed needle		26	26/51/AS	G6500-80118
250	Fixed needle		26	26/51/AS	G6500-80119
	Replacement plunger	10/pk			G4200-80102
500	Fixed needle		26	26/51/AS	G6500-80120
	Replacement plunger				G4200-80106

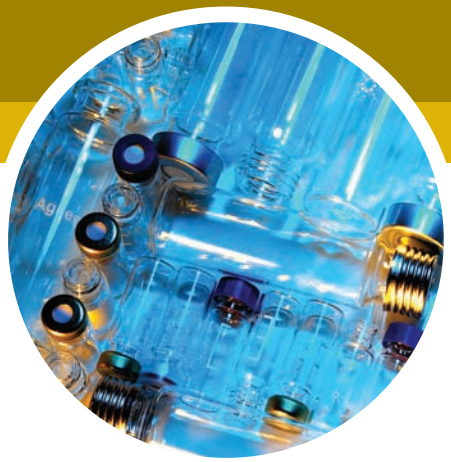
CombiPAL Headspace Supplies

Our fixed-needle headspace syringes feature a sideport needle for gas flushing, in conformance with strict CTC standards. Use with Agilent's Merlin Microseal to minimize instrument downtime – and to prevent lost or compromised data caused by septum leaks and liner contamination.

CTC syringe, G6500-80109

CombiPAL Headspace Syringes

Volume (µL)	Description	Gauge	Part No.
1	Fixed needle	23	G6500-80107
	Replacement plunger		G4200-80101
2.5	Fixed needle	23	G6500-80109
	Replacement plunger		G4200-80107
5	Fixed needle	23	G6500-80111
	Replacement plunger		G4200-80108



CTC recommends screw-top vials and caps for the tightest seal and the most reproducible headspace results, and the precision-thread vials and caps listed are an excellent choice for dependability and ease of use. They are ideal for applications in the environmental, food and beverage, industrial hygiene, drug analysis, and clinical chemistry industries.

CombiPAL Headspace Supplies

Description	Part No.
10 mL, screw top clear vial, 100/pk	5188-5392
20 mL, screw top clear vial, 100/pk	5188-2753
10 mL, screw top amber vial, 100/pk	5188-6538
20 mL, screw top amber vial, 100/pk	5188-6537
UltraClean 18 mm magnetic screw cap with silicone/PTFE septa	5188-2759
Liner, inlet for SPME, deactivated	5188-6471

CombiPAL Autosampler

Stratum PTC Sample Concentrator

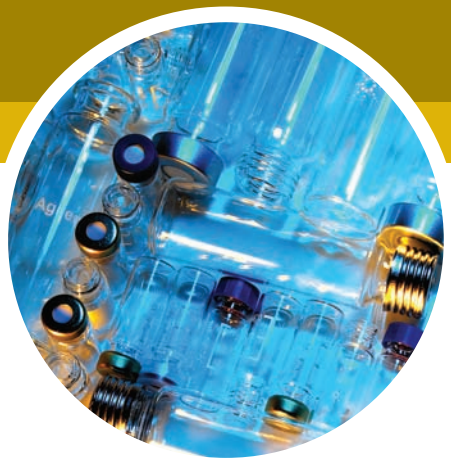
Purge and Trap Supplies

Sparger

Compared to a frit sparger, the fritless sparger may be the better choice when a water sample has a tendency to foam. This sparger is not appropriate for soil samples, which tend to clog the capillary tube. Available in 1/2 and 3/4 in. mount sizes.

Glassware for Teledyne Tekmar Purge and Trap Concentrators, 1/2 in. Mount

Description	Part No.
5 mL frit sparger (glassware only)	5182-0852
5 mL frit sparger kit with fittings	5182-0846
25 mL frit sparger (glassware only)	5182-0851
25 mL frit sparger kit with fittings	5182-0845
5 mL fritless sparger (glassware only)	5182-0850
5 mL fritless sparger kit with fittings	5182-0844
25 mL fritless sparger (glassware only)	5182-0849
25 mL fritless sparger kit with fittings	5182-0796
5 mL needle sparger (glassware only)	5182-0848
5 mL needle sparger kit	5182-0795
25 mL needle sparger (glassware only)	5182-0847
25 mL needle sparger kit	5182-0794



Traps for Teledyne Tekmar Stratum Purge and Trap Concentrator

Description	Part No.
Trap, BTEX + MTBE	5188-8813
Trap (#5),OV-1/Tenax/Silica Gel/Charcoal	5188-8814
Trap (#8), Carbopak B/Carbosieve S-III	5188-8815
Trap (#9), Proprietory	5188-8816
Trap, Tenax/Silica Gel/Carbosieve S-III	5188-8817
Strat-Trap, Tenax/Silica Gel, #2	5188-8818
Strat-Trap,Tenax/Silica Gel/Charcoal, #3	5188-8819
Trap, VOCARB 3000	5188-8820
Trap, VOCARB 4000	5188-8821
Trap, BTEX	5188-8822

Traps for Teledyne Tekmar Velocity Purge and Trap Concentrator

Description	Part No.
Trap, Vocarb 3000 (K Trap)	5182-0775
Trap, Vocarb 4000 (I Trap)	5182-0774
Trap, Tenax (A Trap)	5182-0783
Trap, Tenax/Silica Gel/Charcoal (C Trap)	5182-0781
Trap, BTEX	5182-0773
DryFlow moisture trap	14-8911-003

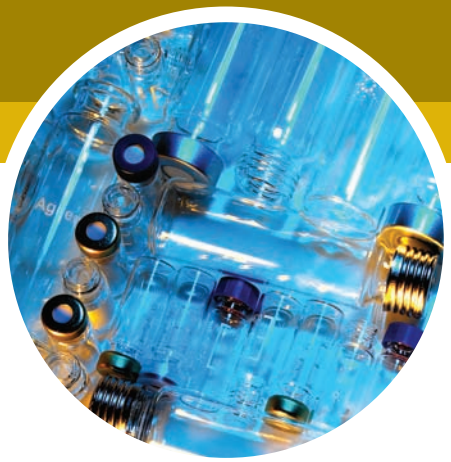
Markes Thermal Desorption

Markes Thermal Desorption system

Agilent now offers a comprehensive line of supplies for Markes Thermal Desorption (TD) instrumentation. Thermal desorption allows the introduction of volatile and semi-volatile compounds from a wide range of sample matrices, directly into a GC or GC/MS.

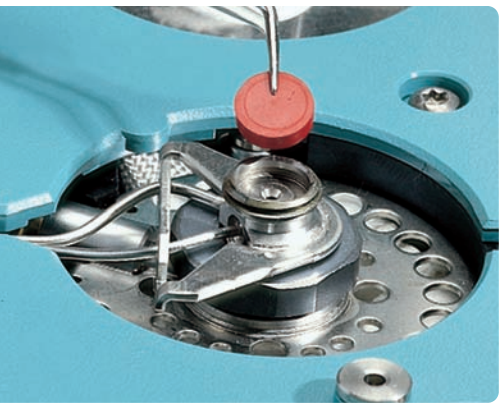
Markes Thermal Desorption Supplies

Description	Unit	Part No.
O-rings, Markes 7 mm cold trap seals	10/pk	MKI-U-COV07
O-rings, Markes 6 mm cold trap seals	10/pk	MKI-U-COV06
PTFE filter disks, 5.1 mm Markes TD	10/pk	MKI-U-DISK1
PTFE filter disks, 6.3 mm Markes TD	10/pk	MKI-U-DISK3
Spare general purpose carbon cold trap		MKI-U-T11GPC
Sampling tube, Tenax TA, Markes Unity		MKI-UTD-5105
Quick fit connectors, Markes Unity	10/pk	MKI-C-QSC10
Stainless steel difflok cap, Markes Unity		MKI-MTD-1169
Silcosteel difflok cap, Markes Unity		MKI-MTD-1204
O-ring insertion tool, Markes Unity TDI		MKI-Z-0285
O-ring extraction tool, Markes Unity TDI		MKI-Z-0351
Cold trap alignment tool, Markes Unity		MKI-UTD-5064
Cold trap, air toxics, C2-C14, Unity 2		MKI-U-T3ATX-2S
Cold trap, air toxics, C2-C14, Unity		MKI-U-T3ATX
Cold trap, materials emissions, Unity		MKI-U-T12ME
Cold trap, GP Carbon, C4/5-C30/32, Unity 2		MKI-U-T11GPC-2S
O-rings, 010 Markes Unity	10/pk	MKI-U-COV10
Cold trap, materials emissions, Unity 2		MKI-U-T12ME-2S
Empty stainless steel TD tubes	10/pk	C-TBE10



Markes Thermal Desorption Supplies

Description	Unit	Part No.
Tenex stainless steel tubes, preconditioned/capped	10/pk	C-TBP1TC
Empty glass TD tubes	10/pk	C-GT010
PTFE inserts	10/pk	C-PL010
Long term TD tube storage caps	10/pk	C-CF020
Cap-LOK Tool for long term storage caps		C-CPLOK
Diffusive sampling caps	10/pk	C-DF010
Bio-VOC breath samplers	10/pk	C-BIO10
Disposable card mouth piece for Bio-VOC	10/pk	C-B010M
Tenax TA 34-60 Mesh, 10 g		C-TNXTA
General purpose hydrophobic tubes, stainless steel Preconditioned and capped with 1/4 in. brass storage caps. For pumped sampling n-C_5 to C_{20}.	10/pk	C-HY010C
Tenax/S'carb 'Sulphur' tubes Preconditioned and capped with 1/4 in. brass storage caps. For odor and landfill gas analysis.	10/pk	C-102SSC
Carbograph 1 stainless steel tubes Preconditioned and capped with 1/4 in. brass storage caps. For pumped C_5-C_{14} plus diffusion of BTX.	10/pk	C-TBP1C1C
Carb X stainless steel tubes Preconditioned and capped with 1/4 in. brass storage caps. For pumped/diffusion 1.3-butadiene & benzene.	10/pk	C-TBP1CXC
Universal stainless steel tubes Preconditioned and capped with 1/4 in. brass storage caps.	10/pk	C-UN010C
Glass tubes with 1 cm Tenax For direct liquid injection.	10/pk	C-G1CM10
Glass air toxics (TO-17) tubes Pre-packed with 2 carbon based sorbents preconditioned and capped with 1/4 in. brass storage caps.	10/pk	C-GAT010C
CRS BTX Standards, 1 µg	10/pk	C-BTX1UG



Inlets

Be certain that you are accurately and predictably introducing samples into your GC and GC/MS Systems.

Proper inlet selection and maintenance can significantly enhance the performance of your chromatography system and, ultimately, your analytical method. It can also prevent problems such as decomposition, flashback, and leaks that can compromise the integrity of your results.

On the following pages, you will find...

- A concise introduction to the selection and use of GC inlets.
- A comprehensive inlet troubleshooting guide.
- A detailed discussion of optimal inlet settings.

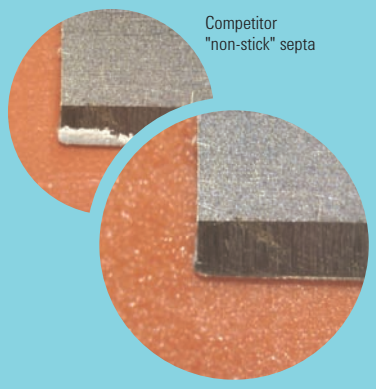
You will also learn how to select the appropriate septa, liners, ferrules, and other inlet supplies.

Premium Non-Stick Septa

Agilent premium non-stick inlet septa are designed and manufactured to provide a reliable non-contaminating seal. Our tri-fold blister pack ensures that each septum remains clean and ready to use.

- Proprietary plasma treatment prevents sticking and unnecessary inlet cleaning.
- Innovative blister package keeps each septum clean and ready for use.
- Center point guides the needle for easy penetration, less coring and longer life.
- Precision molding assures accurate fit in the inlet.
- Each batch is tested on an Agilent 6890 GC-FID for bleed.
- Premium formulations selected for sealing and chromatographic cleanliness.
- No need to bake septa before using.

No Sticking or Clumping



Agilent plasma-treated non-stick septa

Agilent's non-stick septa are plasma treated, eliminating chemical bleed and contamination caused by foreign substances, like the damaging talcum powder used by other suppliers.

Summary of Premium Inlet Septum Characteristics

Septum Type	Bleed	Lifetime	Temperature Limits
Non-Stick BTO (Bleed and Temperature Optimized)	◆◆◆	◆	to 400°C Injection port temp
Non-Stick Long Life	◆	◆◆◆	to 350°C
Non-Stick Advanced Green	◆◆	◆◆	to 350°C

◆◆◆ = *best* ◆◆ = *very good* ◆ = *good*

Bleed and Temperature Optimized Septa (BTO)

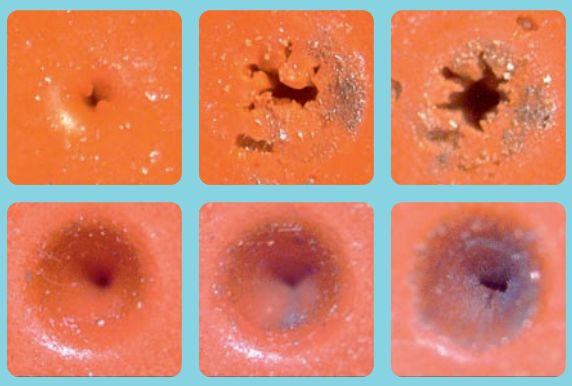
- Extended temperature range, lowest bleed.
- Maximum injection port temperature 400°C.
- Plasma coating eliminates sticking in the injection port.
- Pre-conditioned; ready to use.
- Blister packaging for cleanliness and convenience.
- Ideal for use with low-bleed, "Mass Spec" capillary columns.

BTO septa, 5183-4757

Bleed and Temperature Optimized Septa (BTO)

Description	Unit	Part No.
11 mm septa	50/pk	5183-4757
11 mm septa	100/pk	5183-4757-100
5 mm septa through-hole for on-column, in glass jar	50/pk	5183-4758

Comparison of Coring, With and Without CenterGuide (30x magnification)



High-Temperature Septa without CenterGuide
Major coring before 100 autoinjections

Agilent BTO Septa with CenterGuide
Very little coring even after 700 autoinjections

Advanced Green Septa

- True long-life, high temperature green septum.
- More injections per septum.
- Plasma coating eliminates sticking in the injection port.
- Maximum injection port temperature 350°C.
- High-performance alternative to competitors' "Green" septa.
- Blister packaging for cleanliness and convenience.

Advanced green septa, 5183-4759

Advanced Green Septa

Description	Unit	Part No.
Advanced Green Septa		
11 mm septa	50/pk	5183-4759
11 mm septa	100/pk	5183-4759-100
5 mm septa through-hole for on-column, in glass jar	50/pk	5183-4760

Long-Life Septa

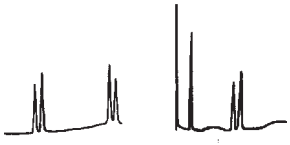
- The preferred septum for autosamplers.
- Pre-pierced for extended life and reduced coring.
- Ideal for overnight runs.
- Up to 400 injections per septum.
- Plasma coating eliminates sticking.
- Maximum injection port temperature 350°C.
- Soft, 45 Durometer, easy on autosampler needles.
- Blister packaging for cleanliness and convenience.

Long-life septa, 5183-4761

Long-Life Septa

Description	Unit	Part No.
11 mm septa	50/pk	5183-4761
11 mm septa	100/pk	5183-4761-100
5 mm septa through-hole for on-column, in glass jar	50/pk	5183-4762

Septa Troubleshooting

Symptom	Possible Causes	Remedy
Extra Peaks/Humps	Septum bleed	Turn off injector heater. If extra peaks disappear, use septum specified for higher temperature or analyze at lower inlet temperature.
		
Baseline Change After Large Peak	Large leak at septum during injection and for a short time thereafter (common with large diameter needles)	Replace septum and use smaller diameter needles.
Retention Times Prolonged	Carrier gas leaks at septum or column connection	Check for leaks. Replace septum or tighten connections if necessary.

Ferrules

Using the wrong ferrule or a worn-out ferrule to seal your column connection can result in inconsistent and unreliable chromatography. An improper ferrule can cause leaks which allow air and other contaminants to enter the instrument through the column seal, causing major interference with column and detector performance.

For optimum performance, ferrules should be replaced every time the column is replaced and when performing column maintenance.

Vespel/Graphite ferrules, 5181-3323

To minimize problems, follow these general techniques for ferrule installation:

- Don't overtighten – finger tighten the column nut, then use wrench to tighten.
- Maintain cleanliness.
- Bake out ferrules prior to use (Vespel and Vespel/Graphite only).
- Avoid contamination, such as fingerprint oils.
- Inspect used ferrules with magnifier for cracks, chips, or other damage before reusing them.
- Change ferrules when new columns or injector/detector parts are installed.

Tips & Tools

Look for the following signals that indicate ferrule damage:

- Background noise from oxygen diffusing into the system
- Column bleed catalyzed by oxygen
- Sample degradation
- Sample loss
- Increase in detector signal/noise
- Poor retention time reproducibility

Ferrule Selection Recommendations

Ferrule/ Seal Type	Upper Temp. Limit	Usages	Advantages	Limitations
Graphite (100%)	450°C	• General purpose for capillary columns. • Suitable for FID and NPD. • Recommended for high temperature and cool on-column applications.	• Easy to use stable seal. • Higher temperature limit. • Can be removed easily.	• Not for MS or oxygen sensitive detectors. • Soft, easily deformed or destroyed. • Possible system contamination.
Vespel/Graphite (85%/15%)	350°C	• General purpose for capillary columns. • Recommended for MS and oxygen sensitive detectors. • Most reliable leak-free connection.	• Mechanically robust. • Long lifetime.	• Not reusable. • Flows at elevated temperature. • Must retighten frequently.
Vespel (100%)	280°C	• Isothermal operation. • Can be reused or removed easily. • Excellent sealing material when making metal or glass connections.	• Mechanically robust. • Long lifetime. • Can be reused or removed easily.	• Leaks after temperature cycle. • Flows at elevated temperature. • Must retighten frequently.
SilTite (100% metal)	N/A	• Use with Capillary Flow Technology nut kits	• Provide leak-tight seals with Ultimate Union, Deans Switch and Effluent Splitter	• Not reusable.

Tips & Tools

When using Vespel/Graphite ferrules, tighten the column nut 1/4 turn after the first temperature program runs. Even preconditioned ferrules can exhibit some shrinkage after a temperature programmed run.

Graphite ferrules, 5080-8853

Capillary Column Ferrules

Ferrule ID (mm)	Column ID (mm)	Unit	Part No.
General Purpose Graphite Ferrules (Short)			
0.5	0.1, 0.2, 0.25, 0.32	10/pk	5080-8853
0.4	0.05, 0.25	10/pk	500-2114
0.8	0.45, 0.53	10/pk	500-2118
1.0	0.53	10/pk	5080-8773
85% Vespel, 15% Graphite Ferrules (Short)			
0.4	0.1, 0.2, 0.25	10/pk	5181-3323
0.5	0.32	10/pk	5062-3514
0.8	0.45, 0.53	10/pk	5062-3512
Preconditioned 85% Vespel, 15% Graphite Ferrules (Long)*			
0.3	0.1	10/pk	5062-3507
0.4	0.1, 0.2, 0.25	10/pk	5062-3508
0.5	0.32	10/pk	5062-3506
0.8	0.53	10/pk	5062-3538
100% Vespel, High Performance Ferrules (Short)**			
0.4	0.1, 0.2, 0.25	10/pk	5181-3322
0.5	0.32	10/pk	5062-3513
0.8	0.45, 0.53	10/pk	5062-3511
Specialty Ferrules, 85% Vespel, 15% Graphite			
Two Hole			
0.4	0.1, 0.2, 0.25	10/pk	5062-3580
0.5	0.32	10/pk	5062-3581
No hole		10/pk	5181-3308
High Temp PTV Inlet, SS/Graphite			
0.4	0.320	10/pk	5188-5315
0.4	0.530	10/pk	5188-5314

**These ferrules are recommended for use with Agilent GC/MS Transferlines with the 05988-20066 MS interface column nut.*

***These ferrules are recommended for use in isothermal analysis only.*

Tips & Tools

100% Vespel ferrules should only be used for isothermal applications.

SilTite Metal Ferrules

Description	Unit	Part No.
For use with 0.25 mm ID capillary columns	10/pk	5188-5361
For use with 0.32 mm ID capillary columns	10/pk	5188-5362
For use with 1/16 in. OD stainless steel tubing Includes 2 column nuts	10/pk	5184-3571
For use with 0.53 mm ID capillary columns	10/pk	5188-5363

Universal column nut, 5181-8830

Column Nuts

Description	Part No.
Short Nuts	
Universal column nut, 1/16 in. hex, 2/pk	5181-8830
Finger tight column nut for 530 µm columns*	5020-8293
Finger tight column nut for 320 µm columns and smaller*	5020-8292
Blanking plug, finger-tight style	5020-8294
6850 column nut, 2/pk	5183-4732
Extended column nut, VI inlet	G3504-20504
High Temp SimDis PTV Inlet, 4 mm hex	5188-5312
Long Nuts	
MS interface column nut, female	05988-20066
Column nut for long or long two-hole ferrules	05921-21170
Accessories	
Open end wrench, 1/4 and 5/16 in.	8710-0510

**For use with graphite ferrules only*

Capillary Flow Technology Supplies

Agilent offers a family of GC accessories based on our proprietary Capillary Flow technology. These accessories increase system productivity and performance:

- QuickSwap MSD interface provides vent-free removal of columns.
- Deans Switch device simplifies the analysis of complex samples.
- Purged Effluent Splitter for inert, leak-free column effluent splitting.

Capillary Flow fittings

Fittings, Ferrules and Supplies

For leak-free, low dead volume and inert column connections with capillary flow accessories, such as the Deans Switch or QuickSwap MSD Interface, use only Siltite ferrules and specified nuts. For Capillary Flow devices, use deactivated fused silica tubing. Do not use tubing that has been coated with stationary phase.

Fittings, Ferrules and Supplies

Description	Part No.
Internal nut	G2855-20530
Swaging nut	G2855-20555
Column storage fitting	G2855-20590
Fused silica, deactivated, 0.15 mm x 1 m	160-2625-1
Fused silica, deactivated, 0.15 mm x 5 m	160-2625-5
Fused silica, deactivated, 0.15 mm x 10 m	160-2625-10
SilTite Metal Ferrules, 0.10-0.25 mm ID capillary columns	5188-5361
SilTite Metal Ferrules, 0.32 mm ID capillary columns	5188-5362
SilTite Metal Ferrules, 0.53 mm ID capillary columns	5188-5363

Split/Splitless Liners

Injection port liners have a variety of features to help vaporize the sample so that a true representation of the sample enters the column. Additionally, Agilent liners are individually packaged to maintain cleanliness until used. The part number and lot are silk screened on the liner for quality control and user convenience, and lot tracking is available for quality assurance.

Dimensions

Agilent liners are made to precise tolerances to ensure GC system accuracy and reproducibility. Controlling the dimensions of the glass provides better consistency from liner to liner, resulting in more reproducible results.

Tips & Tools

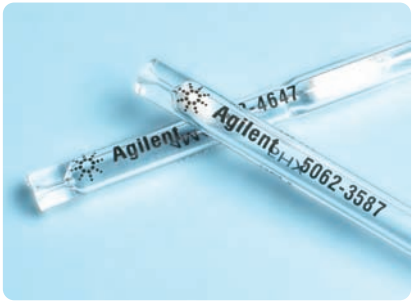
Tight control of liner dimensions is critical to reproducibility of GC results.

Outer Diameter (OD)

Splitless injection liners have a larger OD with a tight fit to improve analyte recovery and limit sample migration out of the liner onto the metal surface of the inlet. Smaller OD liners are used with split injection because there is less resistance to carrier and split flow in the inlet.

Internal Diameter (ID)

Internal Diameter creates the space for the sample to be vaporized. The sample vapor must be small enough to fit within the volume of the liner to avoid backflash, loss of sample into the septum purge, or split lines, which result in reduced reproducibility and sensitivity.



Length

Tight tolerances of liner length are needed to control internal volume and ensure proper sealing between the septum and the inlet seal. Length can also be critical for more repeatable positioning in the inlet relative to the inlet bottom since many analysts install liners by measuring the distance from the O-ring to the top of the liner. Agilent has designed liners with precise glass bumps on the bottom of the liner to automatically position the liner in the inlet for greater reproducibility.

Tapers

None	Bottom Tapers	Dual Tapers
Straight tubes used in split injection with autosamplers.	• Directs sample onto head of column and limits analyte exposure to bottom of inlet. • Minimizes decomposition and discrimination.	• Contain sample within glass liner limiting contact with metal inlet surface. • Thought to limit loss through septum purge.

Splitless liner, double taper, 5181-3315

Glass Wool

- Less discrimination.
- Provides additional surface area for sample vaporization, increasing reproducibility.
- Serves as a trap for non-volatiles.

For split liners, Agilent specifies the placement of glass wool in the liner so that the syringe penetrates the glass wool, wiping the syringe, to provide the most repeatable results with Agilent autosampler and split/splitless inlet design thermal profile.

Even though it is deactivated, glass wool is considered the most active component of a liner, increasing the possibility of adsportion and sample decomposition of active compounds.

Liners with glass wool are not recommended for samples with active analytes – such as phenols, amines, organic acids, pesticides and drugs of abuse – that could be irreversibly adsorbed on surfaces in the inlet.

Deactivation

Agilent's proprietary deactivation is more resilient to degradation than other commercial processes. With use, even deactivated liners become active. Replace the liner regularly.

MS certified split liner, 5188-6576

NEW!

Agilent MS Certified Liners

Agilent MS Certified Split and Splitless Liners are manufactured and tested to our highest level of scrutiny to assure reproducibility.

We have built years of experience into the MS Certified Liners to provide the quality and consistency needed for critical applications, especially those using esterification agents for trace level analysis such as toxicology or drugs of abuse applications.

- Geometrical dimensions and tolerances of the glass are controlled by Statistical Process Control (SPC) with 100% Go-No-Go check.
- Glass wool is pre-qualified with mass spectrometry, then inserted using a unique manufacturing procedure to improve reproducibility.
- Deactivated MS Certified Liners are treated with Agilent's proven proprietary deactivation process developed to last longer than other commercially available treatment.
- Random samples of MS Certified Liners are tested using both FID and MSD analysis of challenging probes to evaluate Acid/Base deactivation, response linearity, peak symmetry, and bleed and background noise.
- Each Agilent MS Certified Liner is traceable by the lot codes silk screened on the liner.

Agilent Split Liners

Agilent Single Taper Split Liners are made to strict dimension specifications for optimal inlet performance and feature the tightest tolerances for OD, ID, taper, and glass wool placement. For ease of use and reproducibility, the liners have a positioning bead, a restriction to secure the position of the glass wool, and a feature to consistently self-position to the recommended height. The liners also feature Agilent's proprietary deactivation.

Agilent recommends part number 5183-4647 as the top split liner choice for:

- Highest run-to-run area reproducibility
- Least discrimination for wide boiling point range samples
- Useable with widest range of conditions and sample types
- Easy self-adjusting installation

Agilent Split Liners

Description	1/pk	5/pk	25/pk
Single Taper Split Liners			
Single taper, glass wool, deactivated, low pressure drop	5183-4647	5183-4701	5183-4702
Single taper, MS certified liner with restriction to hold glass wool	5188-6576		
Single taper, glass wool, deactivated	5183-4711	5183-4712	5183-4713
Straight Split Liners			
Straight, glass wool, non-deactivated	19251-60540	5183-4691	5183-4692
Straight, MS certified liner with glass wool	5188-6574	5188-6569	

Agilent splitless liners

Agilent Splitless Liners

Agilent's proprietary deactivation is important for splitless liners because of the longer sample/liner contact time in splitless mode.

When deciding between a liner with or without glass wool, choose a liner without glass wool if your sample contains non-volatiles or analytes within a wide boiling range. For unknown samples, use a liner without glass wool to avoid loss of active or labile compounds. Agilent recommends first trying a liner without glass wool; use glass wool as a second choice.

Agilent Splitless Liners

Description	Part No.
Single taper, deactivated	5181-3316
Single taper, glass wool, deactivated	5062-3587
Single taper, MS certified liner with glass wool	5188-6568
Double taper, deactivated	5181-3315

Direct Connect Liners

Direct Connect liners

Agilent's Direct Connect Liners provide maximum recovery and minimal decomposition of active compounds for methods requiring splitless injection, such as EPA 8270. They are best for relatively clean samples containing active analytes, such as water extracts. The liners directly connect with the column, similar to press-fit connectors, to aid complete transfer of sample onto the column, eliminating the problem of inlet discrimination and further increasing sensitivity.

Direct Connect Liners

Description	Part No.
Single taper, Agilent proprietary deactivation	G1544-80730
Dual taper, Agilent proprietary deactivation	G1544-80700
Single taper, Deactivated, Inert	G1544-80731

Helix Liners

Helix liners

Helix liners approximates the benefit of glass wool without the activity. The deactivated glass spiral gives surface area without glass wool's active sites to vaporize and mix sample before getting onto the column, and prevents sample from splashing onto the bottom of liner and seal. Helix liners are recommended for slower injection speeds.

Helix Liners

Description	Part No.
Helix open ended, deactivated	5188-5396
Helix double taper, deactivated	5188-5398
Helix single taper, deactivated	5188-5397

Split/Splitless Inlets

The combined split/splitless inlet is the most popular inlet for capillary column gas chromatography. Because it can be used in either split or splitless mode, it provides a very effective combination that can cover most analysis requirements.

Split Inlet Troubleshooting

Split inlets are spared from most band-broadening phenomena, since the splitting process generates narrow peaks. Peak broadening or tailing is usually due to:

- Improper column installation.
- Low inlet temperature.
- Low split flow, (<20 mL/min on 6890).
- Inlet and needle discrimination and decomposition.

If your results are inaccurate or inconsistent:

- Check the column and reinstall if necessary.
- Increase inlet temperature by 50°C and compare results.
- Check inlets and needles for wear and replace as necessary.

Tips & Tools

For the most reproducible split injection results, try Agilent's low pressure drop split liner (part number 5183-4647), with built in positioning bead, tight dimension tolerances, glass wool placement and proprietary deactivation.

Splitless Inlet Troubleshooting

Most problems encountered with a splitless injection are related to:

- Incorrect purge time
- Degradation
- Improper focusing
- Inappropriate column temperature
- Flashback

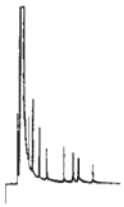
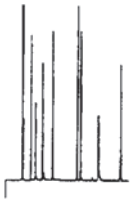
You can also improve the reproducibility and linearity of peak areas and avoid backflash by matching:

- Inlet temperature
- Liner volume
- Injection volume

Decomposition

Loss of peak area or generation of new peaks, can sometimes be dramatically reduced by changing liner type or by deactivating the liner and inlet with silanizing reagents. Removing or reducing the amount of liner packing can also decrease inlet activity.

Column Troubleshooting

		
Normal Peaks	**Tailing Solvent Peaks**	**Wrong Peak Ratios**
Correct column positioning in both injection port and FID	Column positioned incorrectly in the injection port of possible ferrule particle in the carrier gas flow path	Column positioned in the inlet (either too far or not far enough; verify 4-6 mm installation distance)

Split Mode Variables, Practices and Rationales

Parameter	Selection/Setting	Rationale
Inlet temperature	Try 250°C or BP of last eluting compound	Ensures flash vaporization Minimizes inlet discrimination
Inlet liner	Large volume, deactivated	Minimizes flashback Minimizes degradation
Inlet packing	Silanized glass wool Glass beads or frit None	Retains non-volatiles Minimizes inlet discrimination Less active than wool Least active
Injection volume	0.5-3 µL liquid 0.10-10 mL gas	Split easily adjusted Split adjusted accordingly
Injection technique	Fast autoinjection Hot-needle fast manual injection	Less needle discrimination Reproducible discrimination
Split ratio	50:1 to 500:1	Depends on sample and injection volume, and column ID
Initial column temperatures	Not critical	Narrow initial peaks
Septum purge	2-3 mL/min	Minimizes ghosting

Split liner, 5183-4647

Splitless Mode Variables, Practices and Rationales

Parameter	Selection/Setting	Rationale
Inlet temperature	Just above highest boiling point of solutes (+20°C)	Ensures flash vaporization Reduce if degradation occurs Use higher for dirty samples and higher-boiling solutes
Inlet liner	Large volume > 0.8 mL Small volume < 0.2 mL	Use with autoinjector Use only for slow manual injections and gas injections
Inlet packing	None	Use only with slow injection Decreases degradation
	Silanized glass wool	Use for fast autoinjection and dirty samples
Injection volume	0.5 - 2 µL liquid	Depends on solvent, liner and conditions
Injection technique	Fast autoinjection Hot-needle slow manual Hot-needle fast manual	Most reproducible Less needle discrimination Inject 1 - 2 µL/sec if narrow liner is used and > 1 µL injection Use for < 1 µL injections
Purge flow	20 - 50 mL/min	Higher if using constant flow
Purge delay time	20 - 80 sec	Adjust according to column flow rate/liner type and sample conditions
Oven temperature	10 - 25°C below solvent BP	Necessary for solvent focusing
Column flow	> 2 mL/min when possible	Clears inlet fast Reduces backflash and decomposition
Septum purge	2 - 3 mL/min	Reduces ghosting
Quantification	Internal standard External standard addition	Maximizes reproducibility Use only with constant injection volume
Retention gap	1 - 3 m, deactivated (1 - 2 m per µL injected)	Reduces peak distortion Promotes solvent and stationary phase focusing

Flip Top Inlet Sealing System

Agilent's Flip Top Inlet Sealing System is the faster, smarter way to change inlet liners on Agilent GC's.

- Cuts liner replacement time to as little as 30 seconds.
- Eliminates frustrating searches for special wrenches or tools.
- Improves inlet ergonomics – no more handling of heated parts, no more burns or scrapes.
- Decreases downtime and increases productivity.
- Minimizes exposure to ambient air extending column life.
- Installs easily in 15 minutes (customer installable).

Available exclusively from Agilent, the Flip Top has a levered arm that attaches to any 6890/6850/5890 insert weldment and locks to the injection port using an adapter ring screwed onto the inlet. Once installed, the user simply lifts the arm of the Flip Top which releases the insert weldment from the injection port, and allows instant access to the liner. The process is simply reversed to reseal the weldment to the port.

Flip Top Inlet Sealing System

Description	Part No.
Flip Top Inlet Sealing System For 6890, 6850, 5890 only; Not compatible with 7890	5188-2717
Non-Stick Fluorocarbon Liner O-ring for Flip Top, 10/pk	5188-5366

For more information about the Flip Top Inlet Sealing System, visit **www.agilent.com/chem/fliptop**

7890 Turn Top Inlet System

Convenient new turn top design is built into each 7890 split/splitless inlet, allowing you to change liners in less than 30 seconds without special tools or training.

7890 Turn Top Inlet System

Description	Part No.
Turn top	G3430-40035
Split ring	0510-1306
Turn top	G3430-40035

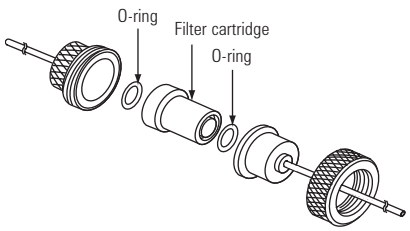
Changing the Split Vent Trap*

1. Remove the retaining clip.
2. Remove the old filter cartridge and two O-rings.
3. Verify the new O-rings are seated properly on the new filter cartridge.
4. Install the new filter cartridge then reassemble the trap. Do not fully tighten yet.
5. Place the filter trap assembly in the mounting bracket and install the retaining clip.
6. Fully tighten the split vent front weldment onto the trap.
7. Check for leaks.

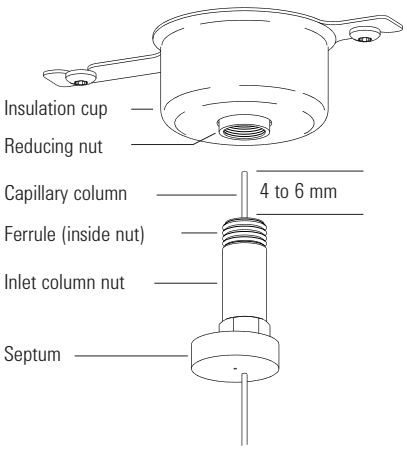
**Change every 6 months*

Warnings & Caution

The split vent trap may contain residual amounts of any samples or other chemicals you have injected into the GC. Follow your company's safety procedures for handling these types of substances while replacing the trap filter cartridge.

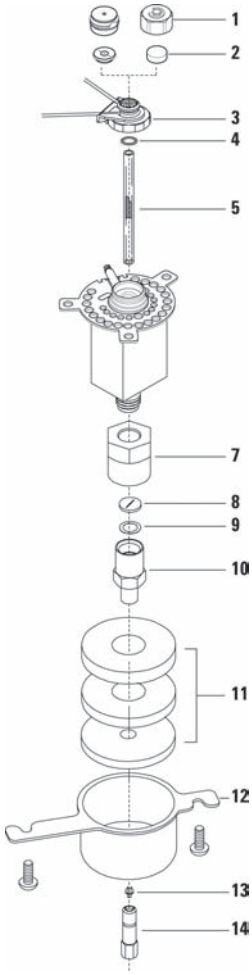


Split vent trap, part number 5188-6495

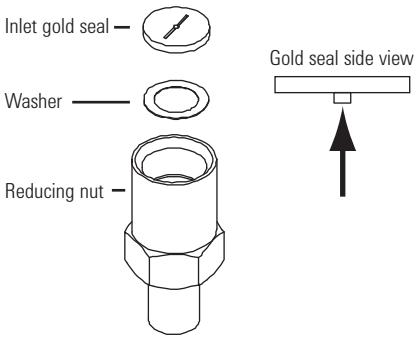


Installing a Capillary Column in a Split/Splitless Inlet

1. Prepare the column for installation.
2. Position the column so it extends 4 to 6 mm past the end of the ferrule.
3. Slide the septum to place the nut and ferrule in the correct position.
4. Insert the column in the inlet.
5. Slide the nut up the column to the inlet base and finger tighten the nut.
6. Adjust the column position so the septum is even with the bottom of the column nut.
7. Tighten the column nut an additional 1/4 to 1/2 turn. The column should not slide with a gentle tug.
8. Start carrier gas flow.
9. Verify flow by submerging the free end of the column in isopropanol. Look for bubbles.



Split/Splitless Inlet assembly



Gold seal on the split/splitless inlet

7890/6890/6850 Split/Splitless Inlet Supplies

Item	Description	Unit	Part No.
	QuickPick Split Vent and Inlet PM Kit		5188-6496
	QuickPick Splitless Vent and Inlet PM Kit		5188-6497
1	Headspace septum retainer nut		18740-60830
	Septum retainer nut		18740-60835
2	11 mm non-stick BTO septa	50/pk	5183-4757
	For complete offering of liners, see pages 58-59		
3	7890 Insert Weldment		
	Top insert assembly, standard		G3452-60730
	Top insert weldment assembly, headspace		G3452-60100
	Top insert, AC gang fitting weldment		G3430-60011
	Top insert assembly, valve		G3480-67585
	6890 Insert Weldment		
	S/SL insert weldment. Used with large charcoal canister type filter, for 6890/6850		G1544-60585
	S/SL insert assembly for G1540A with valved system option. This insert assembly uses the large charcoal canister split vent filter, for 6890/6850		G1580-60585
	Similar to G1544-60575 except carrier lines separated for interface to valved systems of a G1540A instrument		G1580-60575
	Original standard EPC using 1/4 in. split vent filter		G1544-60575
	Similar to G1544-60575 except allowed insertion for 1/4 in. chemical filters to clean carrier gas for ECD operation		G1544-80580
	Insert Weldment Standard manual pneumatics		19251-60575
4	Certified non-stick fluorocarbon O-ring	10/pk	5188-5365
	Graphite O-ring for Split liner	10/pk	5180-4168
	Graphite O-ring for Splitless liner	10/pk	5180-4173
	For complete offering of premium septa, see pages 47-48		
5	Split liner, single taper, low pressure drop, glass wool	1/pk 25/pk	5183-4647 5183-4702
	Splitless Liner, single taper	1/pk 25/pk	5181-3316 5183-4696

7890/6890/6850 Split/Splitless Inlet Supplies

Item	Description	Unit	Part No.
6	Split vent trap kit[1]		G1544-60610
	Replacement cartridge for P/N G1544-60610		G1544-80530
	Split vent trap assembly		G1544-80550
7	Retaining nut		G1544-20590
8	Stainless steel seal		18740-20880
	Certified gold plated seal kit, includes washer[2] Replacement for 18740-20885		5188-5367
	Gold-plated seal with cross[3]		5182-9652
9	Washers, 0.375 OD	12/pk	5061-5869
10	Reducing nut		18740-20800
11	Insulation Kit, 3 pieces		5188-5241
12	Lower insulation cover		19243-00070
13	Ferrules		see page 52
14	Universal column nut	2/pk	5181-8830
	6850 column nut	2/pk	5183-4732
	Split/splitless septum nut angled wrench		19251-00100
	Flip Top Inlet Sealing System For 6890, 6850, 5890 only; Not compatible with 7890		5188-2717
	Capillary Inlet Supplies Kit, Includes:		5181-8838
	Certified gold plated seal kit, includes washer		5188-5367
	Liner, split, straight, glass wool, non-deactivated	4 each*	19251-60540
	Liner, splitless, single-taper, glass wool, deactivated	2 each*	5062-3587
	Certified non-stick fluorocarbon O-ring	10/pk*	5188-5365
	Liner, direct, 2 mm ID, deactivated		5181-8818
	11 mm non-stick BTO septa	50/pk*	5183-4757
	Capillary inlet cleaning wires	5/pk*	5180-4153

[1]Order replacement cartridge G1544-80530 at same time

[2]Use with total inlet flow rates of less than 200 mL/min

[3]Use with total flow rates of greater than 200 mL/min

**Quantity when part ordered individually*

Washers, 5061-5869

Reducing nut, 18740-20800

Certified gold inlet seal, 5188-5367

Cool On-Column Inlets

Cool on-column injection is superior in many ways to other sample introduction techniques. Advantages include:

- Elimination of sample discrimination.
- Elimination of sample alteration.
- Solvent focusing of early eluting solutes.
- High analytical precision.

Sample Considerations

Sample preparation is important for on-column injection because of:

- The potential for column overload, column contamination.
- The incompatibility of some solvents with the stationary phase.
- Dependence of the initial column temperature on the boiling point of the solvent.

Many of the problems associated with these variables can be resolved by using a retention gap ahead of the analytical column.

Troubleshooting

Only columns with an immobilized stationary phase should be used with cool on-column injection as this prevent displacements of the stationary phase by solvents. But problems can still arise due to:

- Column overload.
- Solvent/stationary phase incompatibility.
- Column contamination.

Here are some ways to improve overall performance:

- Prevent broad or split peaks caused by a long flooded zone with a retention gap.
- Use columns with an immobilized stationary phase to prevent displacement of stationary phase by solvents.
- Wash immobilized stationary phases to remove contaminants.
- Use a retention gap for injections of dirty samples.

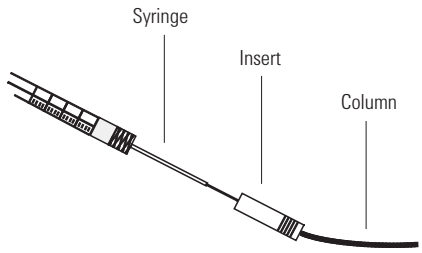
Cool On-Column Inlet Practices and Rationales

Parameter	Selection/Setting	Rationale
Initial inlet temperature	Equal to or 3°C above column oven temperture	Ensures sample focusing in solvent front
Initial inlet temperature ramp	Same as oven (oven track) Faster than oven	Simple and effective Narrows initial peak width
Injection volume	0.1 - 2.0 µL liquid	Use smaller injections for small ID columns Depends on column capacity
Injection technique	Automatically slower injection Fused silica needle	Projects droplets away from syringe tip Use for manual injection into small ID columns
Oven temperature	Inlet temperature or slightly lower	Prevents backflash
Column flow	50 - 80 cm/sec 30 - 50 cm/sec	Use for H_2 carrier gas Use for He carrier gas
Septum purge	12 - 15 mL/min	Use if installed to prevent ghosting
Quantification	All methods	Inherently reproducible technique Lack of discrimination
Retention gap requirements	1 - 3 m, deactivated 530 µm	Corrects peak distortion Protects column from non-volatile components Permits autoinjection with narrow-bore columns

Tips & Tools

There are some limitations to cool on-column inlets, including the following:

- Maximum sample volumes are smaller compared with other inlets (0.5 µL to 2.0 µL).
- Solute peaks eluting just before the solvent cannot be focused and are difficult to determine.
- Capilliary columns (especially those with a large phase ratio or small inner diameter) can be easily overloaded with sample.
- Parameters such as initial column temperature, solvent nature, and injection rate must often be optimized.



Installing a Capillary Column into a Cool On-Column Inlet

1. Gently insert the column into the inlet until it bottoms.
2. Insert the column nut into the inlet fitting and finger tighten.
3. Tighten the column nut an additional 1/4 turn with a wrench or until the column does not move. Use two wrenches to support inlet (5/16 in. and 1/4 in.).
4. If using an automatic injection system with a 0.25 mm or 0.32 mm column, verify that the column installation by manually pushing the syringe into the inlet.

Checking the Needle-to-Column Size on the Cool On-Column Inlet

1. Check the needle-to-column size to make certain that the needle fits in the column.
2. Identify the correct insert for the column size. Use the insert that is the same size as the syringe needle to verify that the column you plan to use is the correct size.
3. Insert the column into one end of the insert.
4. Insert the syringe needle through the other end of the insert and into the column. If the needle cannot pass easily into the column, reverse the insert to try the needle and column in the other end.

Changing the Septum on the Cool On-Column Inlet

1. Replace the septum.

   If you are using a septum nut, grasp the knurling and unscrew. Remove the old septum with tweezers. Use tweezers to install a new septum. Push the septum into the septum nut until properly seated. Firmly tighten the nut.

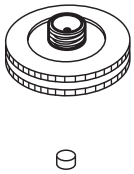
   If you are using a cooling tower, grasp the three rings and unscrew. The spring and duckbill septum may pop out of the inlet when you remove the cooling tower. Be careful not to lose them. If they do not pop out, use a thin wire to remove them from the inlet. Insert the replacement duckbill septum into the spring and place in the inlet. Reattach the cooling tower assembly, then finger tighten.

2. Before making an injection, check the alignment of the entire assembly using the proper size syringe.

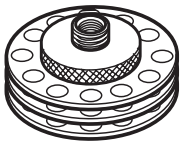
3. Restore the analytical method.

4. Reset the septum counter.

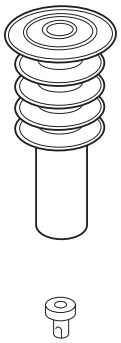
For 250/320-µm automated injections

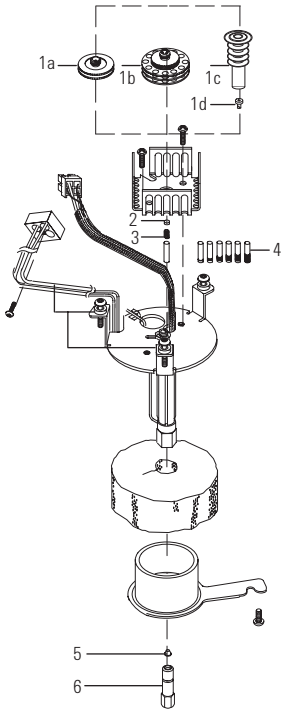


For 530-µm automated injections



For manual 200-µm injections with fused silica needle





Cool On-Column Inlet assembly

7890/6890 Cool On-Column Inlet Supplies

Item	Description	Unit	Part No.
Automatic Injection			
1a	Septum nut base for 320 mm assembly		19245-80521
1b	Septum nut base for 530 mm assembly		G1545-80520
2	Advanced Green Inlet Septa, 5 mm	50/pk	5183-4760
	BTO Inlet Septa , 5 mm	50/pk	5183-4758
Manual Injection			
1c	Cooling tower assembly		19320-80625
1d	Duck bill	10/pk	19245-40050
Fused silica syringe needles		6/pk	19091-63000
On-column syringe, fused silica (barrel only)			9301-0658
Common Supplies			
3	Spring		19245-60760
4	Inserts for capillary columns		
	For 200 µm columns, 1 ring		19245-20510
	For 250 µm columns, 6 rings		19245-20515
	For 320 µm columns, 5 rings		19245-20525
	For 530 µm columns, no rings		19245-20580
	For 530 µm Al clad columns, 4 rings		19245-20780
5	320 µm, 0.5 mm ID graphite ferrule		5080-8853
6	Universal column nut	2/pk	5181-8830

Ultimate Union

Column/Retention Gap Installation Supplies

Description	Unit	Part No.
Ultimate union kit, deactivated		G3182-61580
Ultimate union kit, non-deactivated		G3182-61581
Universal column nut	2/pk	5181-8830
SilTite metal ferrules, 0.10-0.25 mm ID capillary columns	10/pk	5188-5361
SilTite metal ferrules, 0.32 mm ID capillary columns	10/pk	5188-5362
SilTite metal ferrules, 0.53 mm ID capillary columns	10/pk	5188-5363
SilTite metal ferrules for 1/16 in. OD tubing	10/pk	5184-3571
250 µm graphite ferrule	10/pk	500-2114
250 µm retention gap, one 5 m piece		160-2255-5
320 µm retention gap, one 5 m piece		160-2325-5
530 µm retention gap, one 5 m piece		160-2535-5
Deactivated quartz column connector	5/pk	5181-3396

Purged Packed Inlets

Packed column analysis is frequently done when high efficiency separations are not needed or when gases are analyzed by gas-solid chromatography. Purged packed inlets are simple in both design and use. Few parameters need to be set, and all carrier gas flow flushes through the inlet into the column in the standard configuration.

Purged Packed Inlet Practices and Rationales

Parameter	Selection/Setting	Rationale
Inlet temperature	BP of solvent +50°C BP of major solute(s)	Ensures flash vaporization Use for neat samples
Insert type	1/8 in. stainless steel 1/4 in. stainless steel 530 µm	Use for stainless steel column only Inserts permit connection of columns up to 1/4 in. OD
Liner	Glass	Use to lower activity (replaceable)
Initial column temperature	Temperature programming	Sharpens peaks and reduces run time
Column type	1/8 in. packed stainless 1/4 in. packed glass 530 µm	Will not break Better for polar or labile compounds
Carrier gas flow	10-40 mL/min 10-60 mL/min	Use with N_2 carrier gas Use with He or H_2 carrier gas

Purged Packed Inlet Troubleshooting

Purged packed inlets are active, have low volume and are generally flow controlled. This means that most packed column inlet problems involve sample decomposition, flashback, or leaks.

Decomposition

Diagnose inlet sample decomposition by comparing retention times for decomposition products to their standard retention times. Then try these options to improve results:

- Intracolumn direct injection.
- Deactivated glass liners.
- Lower inlet temperatures.
- Remove column packing in the inlet zone.
- Increase flow rates.

Flashback

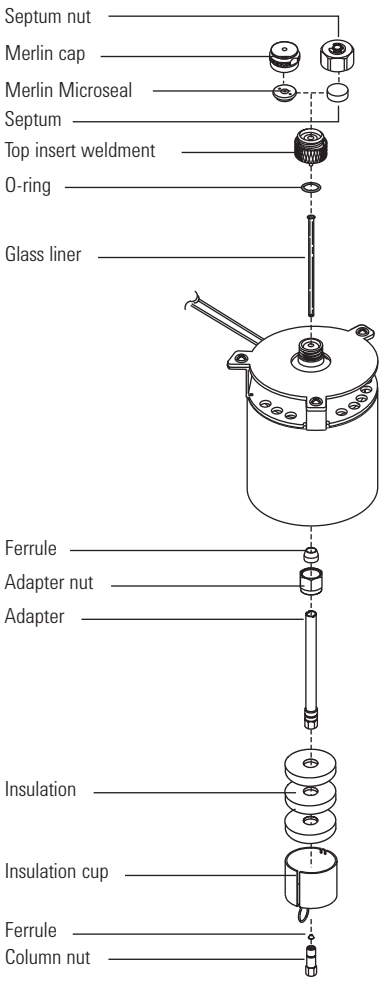
Large sample injections can exceed liner capacity and flash back into the gas supply lines and onto the septum. This can cause:

- Ghost peaks.
- Sample losses.
- Irreproducible peak areas.
- Decomposition.

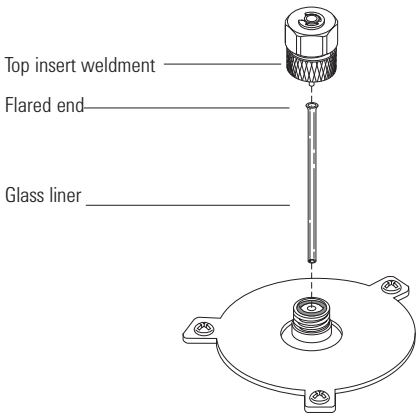
Leaks

Septum and column leaks can cause column degradation and stationary phase decompositions on flow-controlled column inlets.

- Change the septum on a regular basis and check column connections to help eliminate leak holes.
- Keep the oven and inlet at room temperature when not in use or while changing the septum.



Purged Packed Inlet assembly



Glass Liner on Purged Packed Inlet

7890/6890/6850 Purged Packed Inlet Supplies

Description	Unit	Part No.
QuickPick Purged Packed Inlet PM Kit Includes 5 non-stick BTO septa, 1 O-ring, 1 ferrule, and 1 disposable glass liner		5188-6498
Merlin Microseal		5182-3444
Microseal high pressure nut		5182-3445
Septum retainer nut		18740-60835
11 mm non-stick BTO septa	50/pk	5183-4757
Top insert weldment		19243-80570
O-ring, Fluorocarbon	12/pk	5080-8898
Disposable glass liner, 170 µL internal volume	25/pk	5080-8732
Disposable glass insert, deactivated	5/pk	5181-3382
Ferrule, 1/4 in. Vespel	10/pk	5080-8774
1/4 in. nut, brass	10/pk	5180-4105
530 µm column adapter for use with glass liners		19244-80540
1/8 in. column adapter for use with glass liners		19243-80530
1/4 in. column adapter for use with glass liners		19243-80540
Insulating cup		19234-60720
Universal column nut	2/pk	5181-8830

Nuts and Ferrules for 1/8 in. Packed Columns

Description	Unit	Part No.
1/8 in. stainless steel nut and ferrule set	20/pk	5080-8751
1/8 in. brass nut and ferrule set	20/pk	5080-8750
Vespel/graphite ferrule, 1/8 in.	10/pk	0100-1332

Programmed Temperature Vaporizer (PTV) Inlets

PTV inlets combine the benefits of split, splitless and on-column inlets. The sample is usually injected into a cool liner, so syringe needle discrimination does not occur. Then the inlet temperature is increased to vaporize the sample. The user programs vent times and temperature to achieve the equivalent of split or splitless transfer of sample vapors to the column. PTV injection is considered the most universal sample introduction system because of its flexibility.

Advantages

- No syringe-needle discrimination
- Minimal inlet discrimination
- Use of large injection volumes
- Removal of solvent and low boiling components
- Trapping of nonvolatile components in liner
- Split or splitless operation
- Retention time and area reproducibility approaching cool on-column injection

PTV inlets are actively cooled before and during injection by Peltier devices or by forced gases (air, liquid N_2, or liquid CO_2). Cryogenic cooling of the inlet can reduce inlet temperature enough to thermally focus gas injections from other sampling devices in the liner. This is a distinct advantage of using PTV inlets in comparison to conventional inlets for coupling auxiliary sampling devices to capillary columns.

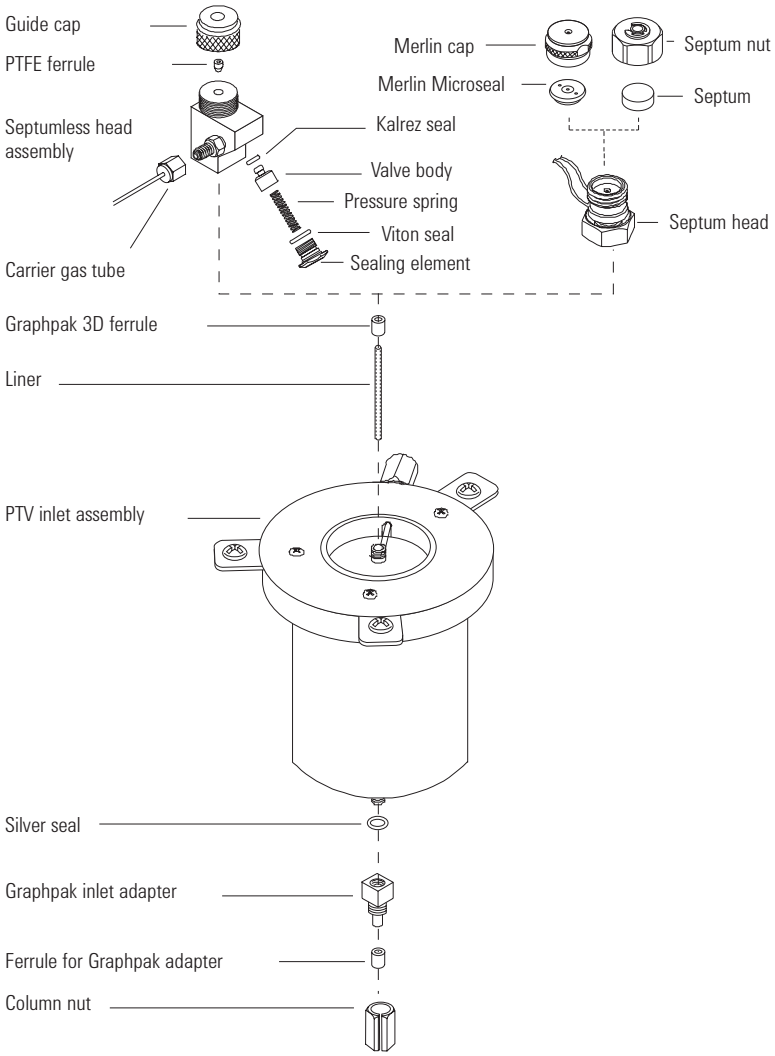
Post-injection, PTV inlets are heated using electrical heaters or preheated compressed air. Depending on design, inlet temperature ramps are either ballistic (i.e., ramped to the maximum temperature at an uncontrolled maximum rate) or programmable.

PTV Inlet Practices and Rationales (cold split/splitless modes)

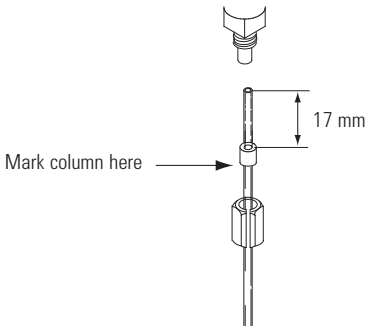
Parameter	Selection/Setting	Rationale
Injection mode	Cold split Cold splitless Cold solvent vent	For general use and sample screening For trace analysis LVI
Inlet temperature ramp rate	Adjustable (i.e., 2°C/sec to 720°C/sec max)	Use slower ramp rates for labile, complex, or large volume samples Use faster ramp rates for most samples Use faster ramp rates to shorten splitless purge delay time
	Ballistic	Simpler, less expensive instrumentation
Inlet liner	Straight with silanized wool Baffled Packed with an adsorbent	For general use For labile samples For focusing gaseous injections from auxiliary sampling devices
Injection volume	0.1 - 1.5 µL 5 - 50 µL for LVI	Use lower volumes for volatile solvents and fast ramp rates Use volumes larger than 1.5 µL only in solvent-elimination mode
Sample Injection technique	Autosampler or manual, fast or slow	Not critical for cold split and splitless modes
Oven temperature	10 - 25°C below solvent BP Sample dependent	For proper solvent effect in splitless mode For split mode
Column flow	30 - 50 cm/sec	Clears inlet faster Less backflash
Septum purge	1 - 5 mL/min	Minimizes ghosting
Quantification	Any method	Inherently reproducible Low discrimination in cold injection modes
Retention gap	1 - 3 m, deactivated	Compensates for extended flooded zone and solvent-column incompatibility

7890/6890 PTV Inlet Supplies

Description	Column ID (mm)	Unit	Part No.
Microseal high pressure nut			5182-3445
Merlin Microseal			5182-3444
Septumless head			G2617-60507
Septum head			G2618-80500
Septum retainer nut			18740-60835
PTV inlet assembly			G2617-60506
PTV LCO_2 cooling jacket			G2617-60508
PTV LN_2 cooling jacket			G2619-60501
Silver seal		5/pk	5182-9763
Graphpack 2M inlet adapter	0.20		5182-9754
	0.25-0.33		5182-9761
	0.53		5182-9762
Ferrules for Graphpak 2M inlet	0.20		5182-9756
	0.25		5182-9768
	0.32		5182-9769
	0.53		5182-9770
Replacement Graphpak column nut			5062-3525
PTV insulation block			G2617-20510
PTV Cryo insulator			G2617-60510
Teflon ferrule (needle seal)		10/pk	5182-9748
Kalrez seal			5182-9759
Valve body			5182-9757
Pressure spring			5182-9758
Viton seal		5/pk	5182-9775
Sealing element			5182-9760
CO_2 Cryo inline filter			3150-0602
Service kit for septumless head Contains Kalrez seal, valve body, and pressure spring			5182-9747
Graphpak 3D ferrules		5/pk	5182-9749
Assembly tool for Graphpack 3D ferrules			G2617-80540



Programmable Temperatue Vaporizing (PTV) Inlet assembly



Installing a Capillary Column into the PTV Inlet

1. Position the column so it extends 17 mm above the end of the ferrule. Mark the column behind the ferrule with correction fluid or a marker. Slide the nut over the column.
2. Insert the column into the adapter and finger tighten the column nut. Looking through the slot in the nut, adjust the column until the mark is correctly positioned below the Graphpak 2M ferrule.
3. Tighten the column nut an additional 1/8 to 1/4 turn with a wrench. Do not overtighten.

Programmable Temperature Vaporizing (PTV) Liners

Description	ID (mm)	Volume (µL)	Part No.
PTV liner, single baffle, glass wool, deactivated	2	180	5183-2038
PTV liner, single baffle, deactivated	2	200	5183-2036
PTV liner, multi baffled, deactivated	1.8	150	5183-2037
PTV liner, high temperature, quartz	3.4	713	5188-5313
PTV liner, high temperature, borosilicate	3.4	668	5188-5356
PTV liner, sintered glass, deactivated	1.5	112	5190-1426

Syringes for PTV Inlet

Volume (µL)	Description	Needle	Part No.
5	Straight, fixed	23/42/HP	9301-0892
10	Straight, fixed	23/42/HP	9301-0713
50	Straight, fixed, for large volume injections	23/42/HP	5183-0318
100	Straight, fixed, for large volume injections	23/42/HP	5183-2058

Detectors

Make sure damaging contaminants do not compromise your GC detector's operation.

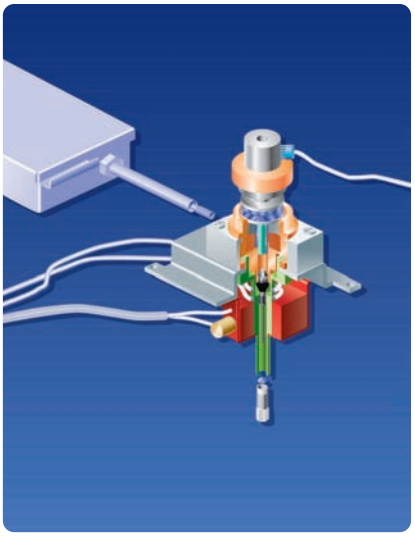
At Agilent, we understand that sensitive GC detectors require regular maintenance and decontamination to ensure peak performance and minimize the risk of costly downtime.

That is why we have devoted this section to...

- The most essential FID, TCD, NPD, ECD, and FPD maintenance and testing procedures.
- Strategies for specific operations, such as testing electron capture detectors for radioactivity leaks and resolving ignition problems associated with your flame photometric detector.
- Techniques for maintaining your detector's high level of selectivity and sensitivity.

Flame Ionization Detector (FID)

The FID requires routine maintenance to ensure optimum performance. Maintenance requirements are application dependent, but Agilent recommends periodically cleaning or replacing the following items:



FID Routine Maintenance

Item	Comments
FID Jet	A plugged jet results in longer retention times as the column exit/detector pressure increases. Once the jet becomes completely plugged, it is difficult to light or sustain a flame.
Igniter Glow-Plug	Replace if corroded or burned out.
FID Collector/Insulators	Contamination can contribute to detector noise or loss of sensitivity.
Column Adapter/Seals For Adaptable FID only	Leaks at column fittings can result in difficulty lighting the FID or sustaining a flame after injection.

Typical FID Problems

Condensation

Since the FID combustion process results in water formation, the detector temperature must be kept above 300°C to prevent condensation. At detector block temperatures below 300°C, the castle assembly drops below 100°C, resulting in condensation and possible rusting. Such condensation, especially when combined with chlorinated or fluorinated solvents or samples, causes corrosion, with resulting increase in detector noise and loss of sensitivity.

FID collector assembly

Flame Ignition

If the flame goes out or will not light:

- Measure the hydrogen/air and makeup flow rates – Low H_2 or makeup flows indicated a plugged jet, or a leak at the column fitting. Measure each gas flow independently.
- Confirm that the igniter is glowing during the FID ignition sequence.
- Check for partially or completely plugged jet – Formation of silica or carbon deposits at the tip of the jet can cause plugging. Incorrect capillary column installation can also cause plugging.

It is best to replace a plugged jet, rather than try to clean it.

- Check that the capillary column is not installed all the way to the jet tip (withdraw 1-2 mm).
- Check that the correct type of jet is installed for the column you are using.
- Check for leaking column or adapter fitting at the base of the FID.
- Check the lit offset value to make sure it is not too low or too high. Adjust the value (normally set to 2.0 pA).

Injecting large volumes of aromatic solvent or water can cause the flame to go out. Switch to a non-aromatic solvent or reduce injection volume.

Tips & Tools

For optimal sensitivity, use Agilent gas purifiers to ensure cleanliness of your GC gases. See page 4.

Increased FID Noise or Loss in Sensitivity

FID noise is affected by:

- The cleanliness of the GC gases and gas delivery system – Ensure that the carrier/H2 and air purity is ≥ 99.9995%. Check traps and filters in the gas supply lines. The FID background signal should be ≤ 20 pA when the flame is lit and stablized.
- Dirty collector/PTFE insulators – Clean or replace.
- Dirty jet – An incorrect flame pattern can increase noise or affect sensitivity.

Installing a Capillary Column in the FID

1. Gather the required supplies and tools.
2. Load the GC maintenance method and wait for the GC to become ready.
3. If using the adaptable detector, verify that the adapter is installed.
4. Place a septum, capillary column nut, and ferrule on the column.
5. Score the column using a glass scribing tool. The score must be square to ensure a clean break.
6. Break off the column end by supporting it against the column cutter opposite the scribe. Inspect the end with a magnifying loupe to make certain there are no burrs or jagged edges.
7. Wipe the column walls with a tissue dampened with isopropanol to remove fingerprints and dust.
8. Install the capillary column.

   If the column ID is greater than 0.1 mm:

   a. Gently insert the column into the detector until it bottoms; do not attempt to force it further.

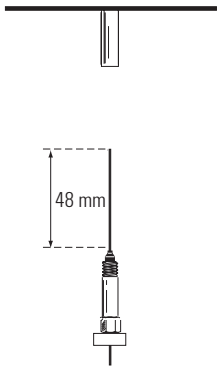
   b. Finger-tighten the column nut, then withdraw the column about 1 mm. Tighten the nut an additional 1/4 turn with a wrench.
   If the column ID is 0.1 mm or less, position the column so it extends above the ferrule by 48 mm (capillary optimized fitting) or 68 mm (adaptable fitting). Slide the septum up to hold the column nut and ferrule at this fixed position.

   c. Insert the column into the detector. Slide the nut and ferrule up the column to the detector base. Finger-tighten the column nut until it grips the column.

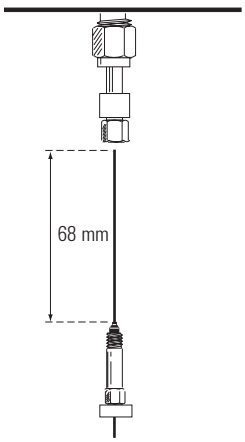
   d. Adjust the column (not the septum) position so that the septum is even with the bottom of the column nut. Tighten the nut an additional 1/4 turn with a wrench.

Positioning the column

Capillary optimized fitting



Adaptable fitting



What you need:

- Column
- Ferrule(s)
- Column nut
- Column cutter
- 1/4 in. open end wrench
- Septum
- Isopropanol
- Lab tissue
- Lint-free gloves

Warnings & Caution

- The oven and/or inlet may be hot enough to cause burns. If either is hot, wear heat-resistant gloves to protect your hands.
- Wear safety glasses to protect your eyes from flying particles while handling, cutting or installing glass or fused silica capillary columns. Use care in handling these columns to prevent puncture wounds.
- Wear clean, lint-free gloves to prevent contamination of parts with dirt and skin oils.

FID Jet Identification and Selection

Before ordering parts for FID maintenance, determine which type of FID is installed on your GC. The FID is available in two versions:

- Dedicated, Capillary Optimized: for capillary columns only
- Adaptable: for packed or capillary columns

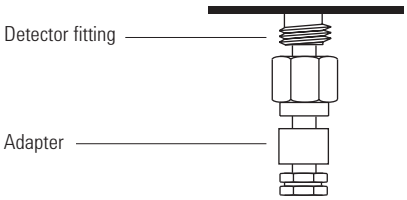
To determine the type of FID installed on your GC, open the oven door and examine the fitting at the base of the detector. Compare to the following diagram.

Hint: Adaptable jets are longer than dedicated capillary jets.

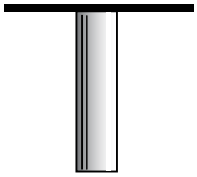
FID Jets

Description	Jet Tip ID	Length (mm)	Part No.
Jets for capillary optimized fittings			
Capillary	0.29 mm (0.011 in.)	48	G1531-80560
Capillary, high temperature Use with simulated distillation	0.47 mm (0.018 in.)	48	G1531-80620
Jets for adaptable fittings			
Capillary	0.29 mm (0.011 in.)	61.5	19244-80560
Capillary, high temperature Use with simulated distillation	0.47 mm (0.018 in.)	61.5	19244-80620
Packed	0.46 mm (0.018 in.)	63.5	18710-20119
Packed, wide-bore Use with high-bleed applications	0.76 mm (0.030 in.)	63.5	18789-80070

Adaptable fitting



Capillary optimized fitting



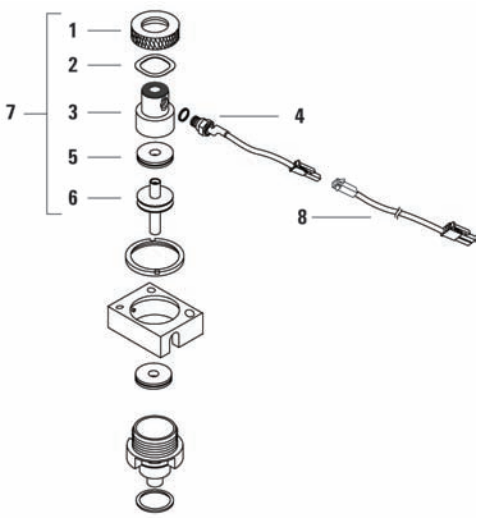
Jet Cleaning Procedure

Use Agilent cleaning kit, part number 9301-0985

1. Run a cleaning wire through the top of the jet. Run it back and forth a few times until it runs smoothly. Be careful not to scratch the jet. (Do not force too large a wire or probe into the jet opening or the opening will become distorted. A loss of sensitivity, poor peak shape, and/or lighting difficulties may result if the opening is deformed.)
2. Fill an ultrasonic cleaning bath with aqueous detergent, and place the jet in the bath. Sonicate for five minutes.
3. Use a jet reamer to clean the inside of the jet.
4. Sonicate again for five minutes.

   Note: from this point on, handle the parts only with forceps!
5. Remove the jet from the bath and rinse it thoroughly, first with hot tap water and then with a small amount of GC-grade methanol.
6. Blow the jet dry with a burst of compressed air or nitrogen, and then place the jet on a paper towel and allow to air dry.

FID cleaning kit, 9301-0985



Flame Ionization Detector (FID) assembly

Flame Ionization Detector (FID) Supplies

Item	Description	Unit	Part No.
	PTFE chimney (optional)		19231-21050
1	Collector nut		19231-20940
2	Spring washer	10/pk	5181-3311
3	Ignitor castle		19231-20910
	Hastelloy ignitor castle (optional)		19231-21060
4	Ignitor glow plug assembly		19231-60680
5	Collector insulator		G1531-20700
6	Collector body		G1531-20690
	Hastelloy Collector Body		G1531-21090
7	FID collector assembly		G1531-60690
	FID collector cleaning brush	2/pk	8710-1346
	Collector Housing		G1531-20740
	FID retainer nut wrench 5880, 5890, 6890		19301-00150
	1/4 in. nut driver for FID jet, drilled shaft		8710-1561
8	FID ignitor cable for 6890/6850 only		G1531-60680
	FID ignitor cable, 7890A only		G3431-60680
	FID performance evaluation sample kit This sample is used for the HP 5880, 5890 and 6890 with a FID or TCD. Solution of 0.033% C14, C15, and C16 normal alkanes in hexane. Three 0.5 mL ampoules.		18710-60170

Test samples

Flame Ionization Detector (FID) Supplies

Item	Description	Unit	Part No.
	FID MDL test sample for 7890 only 3 x 0.5 mL ampoules. Contains 2.36 mg/L n-Tridecane, 2.36 mg/L n-Tetradecane, 23.6 mg/mL n-Penta-decane, 23.6 mg/mL n-Hexadecane in iso-octane		5188-5372
	O-rings	12/pk	5080-4978
	FID/NPD adapter for capillary column		19244-80610
	FID/NPD 1/8 in. packed column		19231-80520
	FID/NPD 1/4 in. packed column		19231-80530
	1/4 in. nut driver for FID jet, drilled shaft		8710-1561
	FID collector cleaning brush	2/pk	8710-1346
	FID Supplies Kit, Includes:		5182-3450
	Jet, packed standard 0.018 in. ID tip	3 each	18710-20119
	FID and TCD Sample	2 each	18710-60170
	Ignitor glow plug assembly	2 each	19231-60680
	Jet, 0.011 in. ID tip, capillary adaptable	3 each	19244-80560
	FID flow measuring insert	2 each	19301-60660
	Cleaning wires for 0.03 in. ID jet	5/pk	5180-4150
	Cleaning wire for 0.018 in. ID/530 µm jet	5/pk	5180-4152
	Wire, jet cleaning	5 each	19301-20720
	Capillary inlet cleaning wires	5/pk	5180-4153
	FID cleaning kit		9301-0985

Electron Capture Detector (ECD)

Liner Selection

The only assembly that requires routine maintenance is the glass liner in the makeup gas assembly, especially for the µECD. All sample passes through the indent in the mixing liner of the µECD. The mixing liner should be replaced if there is a significant loss of sensitivity or any time the column is removed/reinstalled in the detector.

- Gigabore Liner (p/n 19233-20625): for original ECD design (5890 and 6890), brown, polyamide coating
- Mixing Liner (p/n G2397-20540): for µECD, clear glass with indent

Makeup Gas Adapter Maintenance/Installation Procedure

1. Remove the Makeup Gas Adapter from the ECD fitting with a 9/16 in. wrench. Be careful not to stress the 1/16 in. stainless steel gas supply tube.
2. Unscrew the end cap of the Make Gas Adapter and ultrasonically clean in solvent.
3. Remove the old liner.
4. Clean the Makeup Gas Adapter body with solvent in a Nalgene squeeze bottle.
5. Wipe the Makeup Gas Adapter with a clean laboratory wipe.
6. Install the replacement liner.
7. Reinstall the tip of the Makeup Gas Adapter and tighten securely.
8. Reinstall the Makeup Gas Adapter. Make sure it is fully inserted into the detector.
9. Reinstall the column.
10. Reinstall the insulation cup.

Agilent's plasma treated inlet O-rings are pre-cleaned using a two-step proprietary process to minimize contaminants common in flurocarbon O-rings. Order part number 5188-5365 for standard split/splitless inlets and 5188-5366 for the Flip Top.

Thermal Cleaning

If your baseline is noisy or the output value is abnormally high (> 1000 Hz), and you have determined that these problems are not being caused by leaks in the GC system, you may have contamination in the detector from column bleed and sample residues. To remove contamination, you should perform a thermal cleaning (bakeout) of the detector. Bakeout the detector at 20-30 degrees higher than normal operating temperature (375°C Max), with 50-100 mL/min of makeup gas flow.

Warning: Detector disassembly and/or cleaning procedures other than thermal should be performed only by personnel trained and licensed appropriately to handle radioactive materials. Trace amounts of radioactive ^{63}Ni may be removed during other procedures, causing possible hazardous exposure to β and x-radiation.

Radioactivity Leak Test

Electron capture detectors must be tested for radioactive leakage at least every six months. Records of tests and results must be maintained for possile inspection by the Nuclear Regulatory Commission and/or the responsible local agency. More frequent tests may be conducted when necessary.

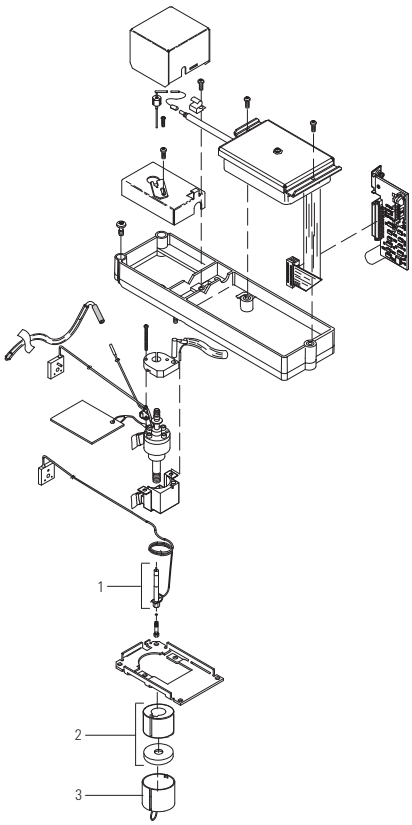
The procedure used is a "wipe test." A wipe test kit is supplied with each new detector. Refer to the information card supplied in the Wipe Test Kit for instructions on performing the test.

Gas Purity

For successful EC detection, it's important that the carrier and purge gases are very clean and dry (99.9995% minimum purity). Moisture, oxygen, or other contaminants can result in higher detector response but usually at the expense of both sensitivity and linear range. Always precondition the column before connection to the detector.

ECD Wipe Test

A wipe test kit (P/N 18713-60050) is supplied with each new ECD. The kit includes an information card with instructions for performing the test. Records of tests and results must be maintained for possible inspection by the Nuclear Regulatory Commission (NRC) and/or responsible state agency.



Electron Capture Detector (ECD) assembly

Electron Capture Detector (ECD) Supplies

Item	Description	Part No.
1	Standard ECD makeup gas adapter*	G1533-80565
	Micro ECD makeup gas adapter	G2397-80520
	Micro ECD mixing liner, also compatible with standard ECD design	G2397-20540
	ECD makeup gas adapter, 7890A only	G3433-67565
2, 3	Insulating cup	19234-60720
	Gigabore liner for standard ECD, polyamide coating, not compatible with micro ECD	19233-20625
	ECD adapter end cap	19233-20755
	Ferrule, 1/4 in. Vespel	5080-8774
	1/4 in. nut, brass	5180-4105
	Electron Capture Detector Sample This sample is used for the HP 5880, 5890 and 6890 with an ECD. Solution of 33 pg/mL (0.033 ppm) (w/v) each of lindane and aldrin in isooctane. Three 0.5 mL ampoules.	18713-60040
	Micro ECD wipe test kit	18713-60050

**Includes one each of P/N 19233-20625 and 19233-20755*

ECD Warnings

Although beta particles at this energy level have little penetrating power – the surface layer of the skin or a few sheets of paper will stop most of them – they may be hazardous if the isotope is ingested or inhaled. For this reason the cell must be handled with care. Radioactive leak tests must be performed at the required intervals, the inlet and outlet fittings must be capped when the detector is not in use, corrosive chemicals must not be introduced into the detector, and the effluent from the detector must be vented outside the laboratory environment.

Thermal Conductivity Detector (TCD)

The TCD compares the thermal conductivities of two gas flows – pure carrier gas (also called the reference gas) and carrier gas plus sample components (also called column effluent).

Filament Maintenance

The primary maintenance for a TCD involves the filament. Most procedures involve improving filament life or keeping the filament from becoming damaged or contaminated. To avoid filament damage and contamination:

- Check for leaks.
- Use gas purifiers to remove oxygen.
- Avoid chemically-active sample components, such as acids and halogenated compounds.
- Turn off the filament when not in use.

Tips & Tools

After maintenance to the gas supply, inlet, or detector, check for leaks with Agilent's Leak Detector (part number G3388A). See page 18.

Increasing Filament Lifetime

Use the following startup process to increase filament lifetime:

Purge the detector with carrier and makeup gas for 10-15 minutes before turning on the filaments. This prevents oxidation of the filaments due to the presence of oxygen that has diffused into the cell under no flow conditions.

Cell Contamination

Cell contamination is a problem when a lower detector temperature is used to improve sensitivity. If the cell becomes contaminated, a solvent flush of the detector may help to remove the condensed material.

Solvent Flush

1. Cool the cell to room temperature and remove the column.
2. Place a septum in a nut or fitting assembly that fits onto the detector entrance (7 mm septum in a 1/8 in. nut).
3. Place the nut or assembly on the detector fitting and tigthen. Verify the presence of makeup gas flow.
4. Inject 20-100 µL volumes of toluene or benzene into the detector through the septum. Inject a total volume of at least 1 mL of solvent. Do not inject halogenated solvents such as methylene chloride and chloroform into the detector.
5. After the final injection, allow makeup gas to flow for 10 minutes or more. Slowly raise the temperature of the cell to 20-30°C above the normal operating temperature.
6. After 30 minutes, decrease the temperature to the normal value and install the column as usual.

Thermal Cleaning

The TCD can become contaminated with deposits from such things as column bleed or dirty samples. A wandering baseline, increased noise level, or changes in response on a checkout chromatogram all indicate contamination. Thermal cleaning, or bakeout (heating the detector block to evaporate the contaminant), should be performed only after you have confirmed that the carrier gas and the flow system components are leak free and contaminant free.

Watch out for decreased sensitivity caused by samples that react with the filament, originating from oxygen-contaminated carrier gas, leaks in plumbing, or column bleeding. Samples with active components, such as acids and halogenated compounds can chemically attack the filament as well. Also, sample condensation will contaminate the detector cell if the temperature is too low.

Some types of contaminants can be removed by temperature bakeout.

Installing a Capillary Column in the TCD

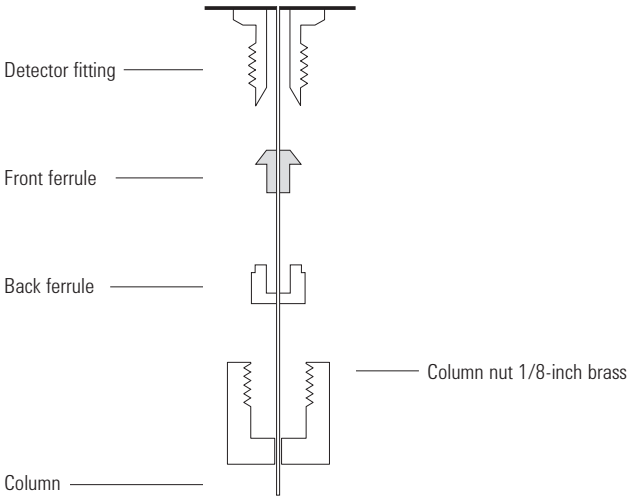
1. Gather the required supplies and tools.
2. Assemble the ferrules and 1/8 in. brass Swagelok nut on the column.
3. Score the column using a glass scribing tool. The score must be square to ensure a clean break.
4. Break off the column end by supporting it against the column cutter opposite the scribe. Inspect the end with a magnifying loupe to make certain that there are no burrs or jagged edges.
5. Wipe the column walls with a tissue dampened with isopropanol to remove fingerprints and dust.
6. Insert the column into the detector until it bottoms.
7. Slide the column nut and ferrules up the column to the detector and finger tighten the nut.
8. Pull out 1 mm of column. Tighten the nut an additional 1/4 turn with a wrench or until the column does not move.

What you need:

- Front ferrule
- Back ferrule
- Column nut
- Column cutter
- 7/16 in. wrench
- Lab tissue
- Lint-free gloves

Warnings & Caution

- The oven and/or inlet may be hot enough to cause burns. If either is hot, wear heat-resistant gloves to protect your hands.
- Wear safety glasses to protect your eyes from flying particles while handling, cutting or installing glass or fused silica capillary columns. Use care in handling these columns to prevent puncture wounds.
- Wear clean, lint-free gloves to prevent contamination of parts with dirt and skin oils.



TCD Ferrules

Column ID (mm)	Back Ferrules	Front Ferrules, 10/pk
0.53	5182-3477	5182-9673
0.32	5182-3477	5182-9676
0.25 / 0.2 / 0.1	5182-3477	5182-9677
No hole	5182-3477	5182-9679
TCD Back Ferrule for 1/8in detector fitting 10/pk	5180-4103	

Determining the TCD Electronic Pressure Control (EPC)

If you have a 6890A or 6890A Plus GC, you may have an older design EPC Flow manifold for the TCD. The older design requires removal of sheet metal panels to attached the TCD reference flow gas supply inside the GC. The new "Minifold" design allows TCD reference gas to be connected directly to the back of the GC. Replacement TCD filament block assemblies have different part numbers depending on the EPC design type.

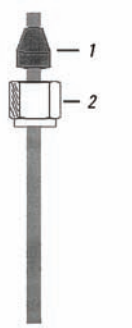
Once you have determined the type of EPC module, decide whether to order a passivated filament block assembly. The passivated assembly is recommended for fatty acid analysis or reactive/acidic samples.

TCD Filament Block Assemblies

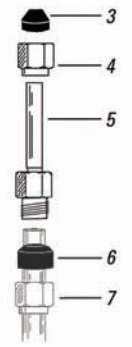
Instrument	Passivated	Applications	Specifications	EPC Design	Part No.
7890A	Yes	Standard TCD Analysis Gases/Hydrocarbons	Filament Block Only Must reuse heater/sensor	Original	G3432-67685
6890	No	Standard TCD Analysis Gases/Hydrocarbons	Filament Block Only Must reuse heater/sensor	Original	G1532-60675
6890	No	Standard TCD Analysis Gases/Hydrocarbons	Filament Block Only Must reuse heater/sensor	Minifold	G1532-60685
6890	Yes	Recommended for Fatty Acid Analysis	Filament Block Only Must reuse heater/sensor	Original	G1532-60690
6890/6850	Yes	Recommended for Fatty Acid Analysis	Filament Block Only Must reuse heater/sensor	Minifold	G1532-60695
6890/6850	No		Complete Detector Assembly Includes detector palette and heater/sensor assembly	Minifold	G2630-61230

Thermal Conductivity Detector (TCD) Supplies

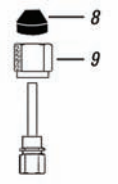
Item	Description	Unit	Part No.
For 1/8 in. SS Packed Column Installation			
1	Vespel/graphite ferrule, 1/8 in.	10/pk	0100-1332
2	1/8 in. nut, brass	10/pk	5180-4103
For 1/4 in. SS Packed Column Installation			
3	Vespel/graphite ferrule, 1/8 in.	10/pk	0100-1332
4	1/8 in. nut, brass	10/pk	5180-4103
5	1/4 in. Packed Column Adapter		G1532-20710
6	Ferrule, 1/4 in. Vespel	10/pk	5080-8774
7	1/4 in. nut, brass	10/pk	5180-4105
For Capillary Column Installation (Standard)			
	TCD Capillary Column Adapter		G1532-80540
8	Vespel/graphite ferrule, 1/8 in.	10/pk	0100-1332
9	1/8 in. nut, brass	10/pk	5180-4103
	Universal column nut	2/pk	5181-8830
	6850 column nut	2/pk	5183-4732
	530 µm, 1.0 mm ID graphite ferrule	10/pk	5080-8773
	320 µm, 0.5 mm ID graphite ferrule	10/pk	5080-8853
	FID and TCD Sample		18710-60170



1/8 in. SS packed column



1/4 in. SS packed column



Standard design

Flame Photometric Detector (FPD)

In 2005, Agilent released an improved FPD with minimum detectable levels (MDL) of 3.6 pg/sec for sulfur and 60 fg/sec for phosphorus. This is more than a 5x improvement for sulfur. The updated design is based on a one-piece, deactivated transferline jet assembly and improved optics. Upgrade kits are available.

Operation

The FPD uses three gases: air and hydrogen to support the flame, and nitrogen makeup for capillary columns. The flow rates are critical for optimizing performance. Using nitrogen as a makeup gas is essential to obtaining low MDLs. Do not use helium for the makeup gas.

Recommended Gas Flows

Detector Gas Flows	Phosphorous Mode	Sulfur Mode
Air	75 mL/min	50 mL/min
Hydrogen	100 mL/min	60 mL/min
Nitrogen makeup	60 mL/min	60 mL/min

Maintenance

Managing gas purity; contamination from column bleed, sample residue, and corrosion; and air leaks can help keep your FPD at peak performance.

Gas purity

Sulfur contamination is a common problem and causes noise and/or a higher baseline offset in the FPD. To minimize sulfur contamination and achieve the lowest MDLs, use at least 99.9995% pure gases, clean tubing, and regulators with metal diagrams. To protect your FPD over its lifetime, Agilent recommends gas generators or supply gas filters designed to remove sulfur.

Glow plug

Contamination

The FPD is susceptible to build up of residue on the surfaces of the igniter coil, jet, combustion chamber, and chamber window. The residue increases detector offset and reduces the signal-to-noise ratio. The sample or column bleed usually cause the residue. After a period of time, you may need to rebuild the detector and replace the transfer line. Do not clean the transfer line, jet, or other parts with brushes or solvents.

To increase the time between servicing, remove the column, cap off the detector, and run it at 250°C with the flame to bake off some of the residue. Replacing the igniter may reduce baseline output. If these tactics are not effective, rebuild the detector.

If your solvent or sample is corrosive, it can erode the aluminum vent tube. Agilent recommends using alternative stainless steel vent tubes for these applications.

For more information about gas purity and selection, including the Renewable Gas Purification System, see pages 3-12.

Air Leaks

The original FPD design has three more internal seals than the new design. Temperature cycling of the detector causes the ferrules to shrink and leaks to occur. The most common leaks are around the fused silica transfer line. To eliminate these leaks, remove the detector from the GC and tighten the transfer line fittings.

For both the original and new FPD, leaks can develop at the column nut or capillary column adapter, the gang fitting at the EPC module, around the vent tube, or around the igniter glow plug. If you are replacing fittings or O-rings, always use conditioned, graphitized-vespel ferrules and Agilent's low sulfur O-rings. Make sure ferrules are the correct size for your column.

Flame Ignition Problems

You can tell if your FPD is lit by checking the detector "Output" and "Flame" on the display. The detector senses that the flame is on by comparing the output with the offset. An optimized FPD normally runs with an output in the range of 30 to 80 with the offset set point at 2.0. If the flame is out and the electrometer is on, the output usually displays less than 1.

Most FPD ignition problems are caused by incorrect gas flows, incorrect column installation, or a dirty or defective igniter. To troubleshoot:

1. Make sure the FPD is at operating temperature before trying to light.
2. Remove the rubber drip tube while lighting the FPD.
3. Increase air supply pressure by 10-20 psi.
4. Check the detector gas flows to see if they match the Recommended Gas Flows table.
5. Check the detector output when you turn the flame on. The photomultiplier will see the glow of the igniter and jump to about 68000 pA.
6. Remove the column and check the tip for residue or burnt polymide coating. If it appears damaged, cut off the damaged portion and reinstall to the proper height.
7. Remove the igniter glow plug. If dirty or damaged, replace it.

Tips & Tools

Helium is not a good makeup gas for the FPD. You will not be able to light or keep the detector lit in the sulfur mode with helium.

Less common problems include leaks, quenching, and condensation:

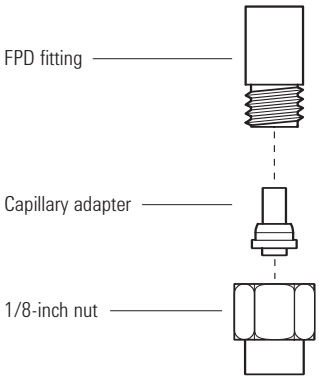
- Large air leaks at the inlet or detector can reduce the percentage of the hydrogen-air mixture at the detector and cause ignition problems.
- Large injections of certain samples can cause flameouts or quenching that cause the detector to attempt to relight, interrupting your analysis.
- Condensation is a by-product of the burning of your sample. For many analyses, the liquid is collected from the vent tube. If the liquid drips back into the detector, it will extinguish the flame. Agilent recommends that you wait to light the flame until the detector is at temperature and equilibrated.
- Light leaks at the vent tube can cause a higher baseline offset. Make sure the vent tube ferrule seals tightly against the emission block. Keep the lid closed over the detector.

Installing a Capillary Column Adapter to the FPD

1. Gather the required supplies and tools.
2. Load the GC maintenance method and wait for the GC to become ready.
3. Insert the capillary adapter into the 1/8-inch nut as shown, then thread the nut onto the detector fitting.
4. Finger-tighten the nut, then tighten an additional 1/8 turn with a wrench.

What you need:

- FPD capillary column adapter
- Column cutter
- 1/4 in. and 9/16 in. wrenches
- Metric ruler
- 1/8 in. nut
- Lint-free gloves



Warnings & Caution

- The oven and/or inlet may be hot enough to cause burns. If either is hot, wear heat-resistant gloves to protect your hands.
- Wear safety glasses to protect your eyes from flying particles while handling, cutting or installing glass or fused silica capillary columns. Use care in handling these columns to prevent puncture wounds.
- Wear clean, lint-free gloves to prevent contamination of parts with dirt and skin oils.

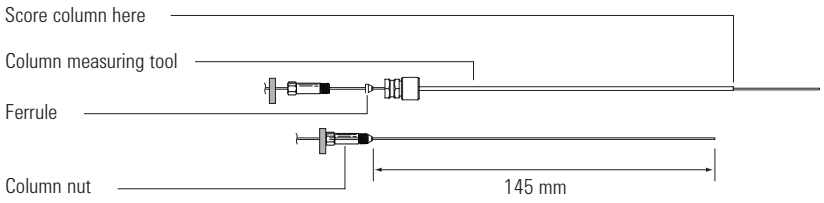
Attaching a Capillary Column to the FPD

What you need:

- Column measuring tool, p/n 19256-80640
- Column cutter
- 1/4 in. and 7/16 in. wrenches
- Column nut
- Ferrule
- Capillary column
- Lint-free gloves

1. Gather the required supplies and tools.
2. Load the GC maintenance method and wait for the GC to become ready.
3. Assemble a septum, column nut, and ferrule on the end of the column.
4. Insert the end of the column through the column measuring tool so that the end protrudes beyond the tool.
5. Tighten the column nut until it grips the column. Tighten the nut an additional 1/8 to 1/4 turn with a pair of wrenches. Snug the septum against the base of the column nut.
6. Use a wafer cutter at 45° to score the column.
7. Snap off the column end. The column may protrude about 1 mm beyond the end of the tool. Inspect the end with a magnifying loupe to make certain that there are no burrs or jagged edges.
8. Remove the column, nut, and swaged ferrule from the tool.
9. Wipe the column walls with a tissue dampened with isopropanol to remove fingerprints and dust.
10. Verify that a capillary adapter is installed in the detector fitting.
11. Carefully thread the swaged column up into the adapter. Finger-tighten the column nut, then use a wrench to tighten an additional 1/8 turn.

If you are using a capillary column, the tip of the column must be at least 1 mm below the surface of the jet. When you install the column, measure the distance from the sealing surface of the ferrule to the tip of the column. This measurement is 153 mm for the original FPD and 145 mm for the new FPD. For the new design, Agilent recommends using the column measuring tool, part number 19256-80640.

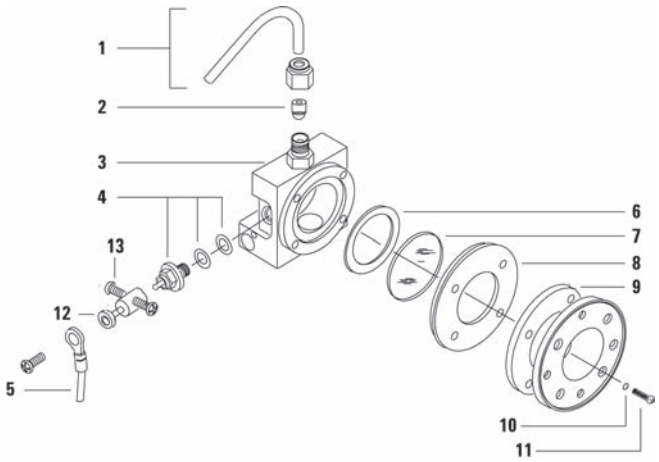


Tips & Tools

Track detector output. When it increases by 50%, remove the column, bake it out, replace the igniter, or rebuild the detector.

FPD Ignitor and Heat Shield Assembly

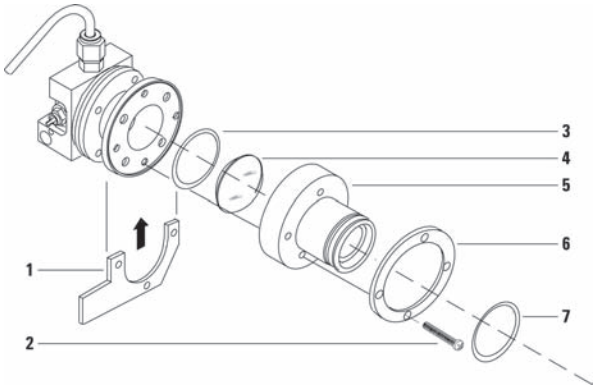
Item	Description	Part No.
1	FPD Exit Tube Assembly	
	Aluminum	19256-60700
	Stainless Steel	19256-20705
2	Ferrule, 1/4 in. Vespel, 10/pk	5080-8774
3	Emission chamber, single	
	FPD single	19256-80560
	FPD, dual	19256-80600
4	FPD igniter replacement kit Includes items A, B, C	19256-60800
	A. O-ring, size 2-010	
	B. Spacer, ignitor	
	C. Glow plug	
5	Igniter cable assembly	G1535-60600
6	Heat shield gasket, white	19256-80045
7	First heat shield window	19256-80030
8	Heat shield disk	19256-20580
9	Stainless steel coupling	19256-20550
10	Lock washer (4 required)	2190-0584
11	Screw, M3 x 12, T10 (4 required)	0515-1084
12	Collar	19256-20690
13	Screw, M3 x 66 mm, T10	0515-0680



FPD ignitor and heat shield assembly

FPD Lens Assembly

Item	Description	Part No.
1	Clamp	19256-00090
2	Screw, M3 x 25 (4 required)	0515-0683
3	Window O-ring, inner, 0.926 in. ID, orange	5061-5886
4	Convex lens	1000-1438
5	Lens housing	19256-20900
6	Flange ring	19256-00200
7	Fluorocarbon Elastomer O-ring, brown, 1.239 in. ID	5061-5890



FPD lens assembly

FPD PMT and Bracket Assemblies

Description	Part No.
Chimney back cover	G1535-80520
Heater/sensor assembly	G1535-60610
Transfer line support bracket	19256-00320
Bracket/support	G1535-00010
Sulfur filter, 7890 and late model 6890*	1000-1437
Sulfur filter, blue, early model 6890*	19256-80000
Phosphorus filter, yellow	19256-80010
Filter spacer (used only with sulfur filter)	19256-20910
PMT housing assembly	19256-60510
Dual FPD chimney front	G1535-00030

**Please contact Agilent technical support for assistance in selecting the correct sulfur filter for your 6890 FPD detector.*

Nitrogen Phosphorus Detector (NPD)

Bead Maintenance

NPDs are temperamental and require frequent maintenance. Small changes in any of a number of parameters can significantly change the performance characteristics of an NPD. The bead requires the most maintenance. It needs to be changed frequently, thus a spare is a necessity.

The beads have to be kept dry which limits their storage life to about six months. When a new bead is installed, slowly raise the detector temperature and bead current. Rapid heating can crack or break the bead especially if it has been stored under humid conditions. It has been observed that higher hydrogen flows and bead currents decrease bead life. If the NPD is not in use, the hydrogen flow and bead current should be reduced or turned off to increase bead life. Make sure there is some type of gas flow in a heated detector or when there is current to the bead.

NPD bead

Bead Life

To extend the life of the bead:

- Avoid polar stationary phase, if possible.
- Use the lowest practical adjust offset or bead voltage.
- Run clean samples and keep the inlet/liner clean to minimize contamination.
- Turn off the bead when not in use.
- Keep the detector temperature high (320-335°C).
- Turn off the hydrogen flow during solvent peaks and between runs.
- If the NPD is off for an extended period of time in a high humidity environment, water may accumulate in the detector. To evaporate this water, set the detector temperature to 100°C and maintain it for 30 minutes. Then set the detector temperature to 150°C and maintain it for another 30 minutes.

Gas Flow

The hydrogen, air and makeup gas flows should be measured frequently. They can drift over time or be changed unintentionally without knowledge of it occurring. Each gas flow should be measured independently to obtain the most accurate values. NPDs are very sensitive to changes in the gas flows and consistent flows are necessary to maintain performance levels.

Measuring NPD Flows

1. Set the bead voltage to 0.0 V.
2. Cool the NPD to 100°C.
3. Remove the bead and store it carefully until re-installation.
4. Insert the NPD flowmeter adapter tool into the NPD collector.
5. Attach the flow-measuring insert to the NPD flowmeter adapter tool.
6. Place the flowmeter tubing over the flow-measuring insert to begin measuring flows.

For more information about gas purity and selection, including the Renewable Gas Purification System, see pages 3-12.

Gas Purity

Because of its high sensitivity, the NPD requires very pure gases (99.999% or better). We strongly recommend that moisture and hydrocarbon traps be used on the carrier gas and all detector gases, including the detector hydrogen, air, and makeup gases. Dirty gases will not only give poor chromatographic performance, but will shorten the bead life as well.

Cleaning and Replacement

The NPD requires periodic cleaning. In most cases, this only involves the collector and the jet. Agilent provides brushes and wires that simplify the cleaning of all detector parts. The brushes are used to dislodge particulates clinging to the metal surfaces. A fine wire is used to clean the jet opening of particulates. Do not force too large a wire or probe into the jet opening or the opening will become distorted. A loss of sensitivity or poor peak shape may result if the opening is deformed. The various parts can be ultrasonicated after cleaning with a brush. Eventually the jet needs to be replaced, so it is strongly recommended to have spare jets on hand.

Over time, residue from the bead or sample can build up in the collector and cause baseline problems. You should clean the collector after you have damaged the bead two or three times.

The metal C-rings wear slightly with each assembly and disassembly. After several assemblies and disassemblies (five or more), the rings may not seal effectively, causing an erratic baseline. A ceramic insulator and seal kit is available. Always cool the detector to near-ambient when changing seals and insulators.

Because there is no flame in the NPD, the jet does not collect silica and soot as does the FID jet. Although you can clean the jet, it is ususally more practical to simply replace dirty jets with new ones. If you do clean the jet, use the cleaning wire, taking care not to damage the inside of the jet. You can also use a sonicator bath to clean the jet.

Contaminants

Some chemical problems can also arise when using the NPD. Because it is a trace detector, be careful not to contaminate the analytical system.

Glassware

Glassware must be very clean. Phosphate detergents should be avoided, so acid washing of glassware followed by distilled water and solvent rinsing is recommended.

Solvents

Solvents should be checked for purity. Chlorinated solvents and silanizing reagents can decrease the useful lifetime of the alkali source; excess reagent should be removed prior to injection, if possible.

Other Contamination Sources

Phosphate-containing leak detectors, phosphoric acid-treated columns or glass wool, polyimide-coated columns, or nitrogen-containing liquid phases can add noise to the system and should be avoided.

NPD Jet Identification and Selection

Before ordering parts for NPD maintenance, determine which type of NPD is installed on your GC. The NPD is available in two versions:

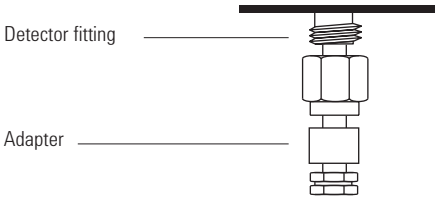
- Dedicated, Capillary Optimized: for capillary columns only
- Adaptable: for packed or capillary columns

Hint: Adaptable jets are longer than dedicated capillary jets.

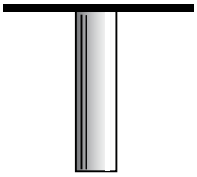
NPD Jets

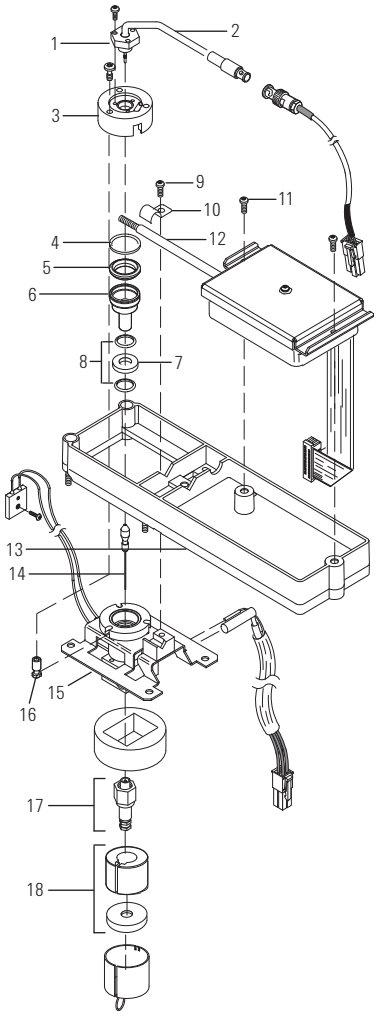
Description	Jet Tip ID	Length (mm)	Part No.
Jets for capillary optimized fittings			
Capillary with extended jet (recommended)	0.29 mm (0.011 in.)	51.5	G1534-80580
Capillary	0.29 mm (0.011 in.)	53	G1531-80560
Capillary, high temperature	0.47 mm (0.018 in.)	48	G1531-80620
Jets for adaptable fittings			
Capillary with extended jet (recommended)	0.29 mm (0.11 inch)	70.5	G1534-80590
Capillary	0.29 mm (0.011 in.)	61.5	19244-80560
Capillary, high temperature	0.47 mm (0.018 in.)	61.5	19244-80620
Packed	0.46 mm (0.018 in.)	63.5	18710-20119

Adaptable fitting



Capillary optimized fitting





Nitrogen Phosphorus Detector (NPD) assembly

Nitrogen Phosphorus Detector (NPD) Supplies

Item	Description	Part No.
1	Screws, M3 x 0.5 x 8 mm (Pozidriv)	0515-0655
2	NPD bead assembly*	G1534-60570
	NPD black ceramic bead assembly**	5183-2007
3	Lid weldment	G1534-80510
4	Metal C-ring, top	0905-2580
5	Alumina insulator, upper	G1534-40020
6	Collector funnel	G1534-20530
7	Alumina insulator, lower	G1534-40030
8	Metal C-ring, bottom	0905-1284
9	Screw, M4 x 07 10 mm	0515-2495
10	J-Clamp	1400-0015
11	Screw, M4 x 07 10 mm	0515-2495
12	NPD interconnect assembly	G1534-60610
13	Mounting pallet	G1531-40020
14	Jet, 0.011 in./0.29 mm ID tip, capillary dedicated	G1531-80560
	Jet, 0.011 in. ID tip, capillary adaptable	19244-80560
	Jet, packed standard 0.018 in. ID tip	18710-20119
15	Base weldment, Capillary NPD for 6890/6850 only	G1534-80500
	Base weldment, Packed NPD for 6890/6850 only	G1534-80540
	Base weldment, Capillary NPD, 7890A	G3434-67500
	Base weldment, Packed NPD, 7890A	G3434-67540
16	Lid stop	G1534-20590
	NPD Ceramic Insulator Kit Includes items 4, 5, 7, and 8	5182-9722
17	FID/NPD adapter for capillary column	19244-80610
	FID/NPD 1/8 in. packed column	19231-80520
	FID/NPD 1/4 in. packed column	19231-80530
18	Insulating cup	19234-60720

**This bead is more sensitive but exhibits some tailing for phosphorous compounds.*

***The black bead is potentially a little less sensitive but does not exhibit peak tailing and typically has longer lifetime.*

Nitrogen Phosphorus Detector (NPD) Supplies

Item	Description	Part No.
	Ferrule, 1/4 in. Vespel, 10/pk	5080-8774
	530 µm, 1.0 mm ID graphite ferrule, 10/pk	5080-8773
	320 µm, 0.5 mm ID graphite ferrule, 10/pk	5080-8853
	1/4 in. nut, brass, 10/pk	5180-4105
	Universal column nut, 2/pk	5181-8830
	Nitrogen-Phosphorus Detector Sample Solution of 0.65 ppm azobenzene, 1,000 ppm octadecane, and 1.00 ppm malathion in isooctane (w/v). Three 0.5 mL ampoules.	18789-60060

Nitrogen Chemiluminescence Detector (NCD)

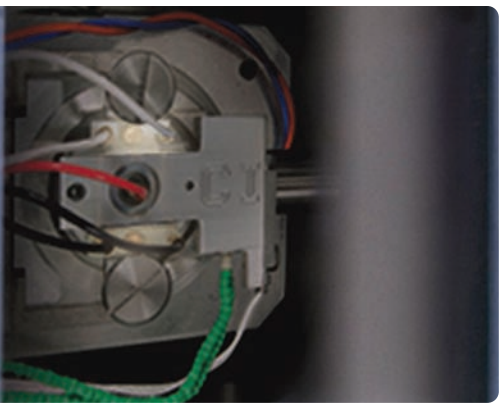
Nitrogen and Sulfur Chemiluminescence Detectors

Nitrogen Chemiluminescence Detector (NCD) Supplies

Description	Part No.
Dual plasma burner accessory kit Includes ferrules, fittings and quartz tube	G6600-60038
PM Kit, dry piston pump Includes dry piston seal, 6 Moleculite vacuum pump traps and 1 set of ceramic tubes	G6600-67006
PM Kit, DP RV5 oil pump Includes 6 chemical traps for Ozone destruction, 3 oil coalescer elements and 4 (1 Qt) bottles of synthetic oil	G6600-67007
PM Kit, dry piston pump Includes 6 Chemical traps for Ozone destruction and 2 repair kits for pump	G6600-67008
12-month maintenance kit Includes 6 quarts oil, 12 chemical traps, 4 oil filter elements, 2 sets of ceramic tubes, and 2 O-rings	G6600-67009
Replacement oil coalescing filter	G6600-80042
Oil mist filter for RV5 pump	G6600-80043
Replacement oil coalescing filter	G6600-80044
Replacement odor filtration element	G6600-80045
O-ring, 1.3614 in. ID	G6600-80050
O-ring, 1.301 in. ID	G6600-80051
Dual plasma quartz tube	G6600-80063
Mobil 1 synthetic oil	G6600-85001
Oil, Edwards ultragrade for RV3 and RV5 pumps	G6600-85002

Sulfur Chemiluminescence Detector (SCD) Supplies

Description	Part No.
PM Kit, dry piston pump Includes dry piston seal, 6 Moleculite vacuum pump traps and 1 set of ceramic tubes	G6600-67006
PM Kit, dry piston pump Includes 6 Chemical traps for Ozone destruction and 2 repair kits for pump	G6600-67008
12-month maintenance kit Includes 6 quarts oil, 12 chemical traps, 4 oil filter elements, 2 sets of ceramic tubes, and 2 O-rings	G6600-67009
Dual plasma burner accessory kit	G6600-60037
Mobil 1 synthetic oil	G6600-85001
Oil mist filter for RV5 pump	G6600-80043
Oil, Edwards ultragrade for RV3 and RV5 pumps	G6600-85002
O-ring, 1.301 in. ID	G6600-80051
Ozone destruction chemical trap	G6600-85000
Replacement oil coalescing filter	G6600-80044
Sulfur Chemiluminescence test sample	G2933-85001
Sulfur trap For carrier H_2 and air gases; one required for each cylinder of gas (3 total)	G2933-85003



GC/MS Systems

Prevent, detect, and correct problems now and promote a lifetime of reliability and low operating costs.

Your mass spectrometer is a sensitive, specialized device that delivers a higher level of functionality than other GC detectors. However, it also requires more stringent adherence to a regular maintenance schedule.

Accordingly, we have dedicated an entire section of this guide to the routine tasks that will help you...

- Prevent contamination.
- Monitor critical functions.
- Maximize your GC/MS system's performance.

We will also show you how to execute key diagnostic tests and choose the right parts and supplies.

Maintaining Mass Selective Detectors (MSD)

Your GC/MSD is highly sensitive. To continue achieving optimal results, it is critical to maintain your system properly by performing the essential tasks within this section. Some of the benefits of maintaining your GC/MSD include:

- Less downtime for repairs.
- Longer lifetime for your MSD system.
- Reduction in overall operating costs.

It is advisable to keep a log book of system performance, Autotune, and maintenance operations performed. This makes it easier to identify variations from normal performance and to take corrective action.

Maintenance Schedule

Task	Every week	Every 6 months	Every year	As needed
Tune the MSD				♦
Change injection port liners	♦			
Check the foreline pump oil level	♦			
Gas ballast the foreline pump	♦			
Check the calibration vial		♦		
Replace the foreline pump oil		♦		
Check the diffusion pump fluid	♦			
Replace the diffusion pump fluid			♦	
Replace the traps and filters			♦	
Clean the ion source				♦
Change the carrier gas trap(s) and purifier				♦
Replace worn out parts				♦
Lubricate seals (where appropriate)				♦
Replace column				♦

MSD Contamination

Contamination is usually identified by excessive background in the mass spectra. It can come from the GC or from the MSD. The source of the contamination can sometimes be determined by identifying the contaminants. Some contaminants are much more likely to originate in the GC, others are likely to originate in the MSD.

Contamination Sources in the GC

- Column or septum bleed
- Dirty injection port
- Injection port liner
- Contaminated syringe
- Poor quality carrier gas
- Dirty carrier gas tubing
- Fingerprints
- Air leaks
- Cleaning solvents and materials

Column bleed generally appears as a continuous and increased rise in the baseline at higher column temperatures, especially at or near the upper temperature limit of the GC column. Septum bleed usually appears as discrete peaks, and can occur at any temperature.

Contamination Sources in the MSD

- Air leaks
- Cleaning solvents and materials
- Fingerprints inside the manifold
- Diffusion pump fluid
- Foreline pump oil

The action required to remove contamination depends on the type and level of contamination. Minor contamination by water or solvents can usually be removed by allowing the system to pump (with a flow of clean carrier gas) overnight. Serious contamination by rough pump oil, diffusion pump fluid or fingerprints is much more difficult to remove and may require extensive cleaning.

Air Leaks

Air leaks are a problem for any instrument that requires a vacuum to operate. Leaks are generally caused by vacuum seals that are damaged or not fastened correctly.

Leaks can occur in other places in the MSD, including the following:

- GC/MSD interface column nut
- Side/top plate O-ring (all the way around)
- Vent valve O-ring
- Calibration valve
- High vacuum gauge tube/controller fitting
- Cracked ion gauge tube (5973/5972/5971)
- Front and rear end plate O-rings
- GC/MSD interface O-ring (where the interface attaches to the vacuum manifold)
- Diffusion pump co-seal
- Baffle adapter O-ring
- Turbomolecular pump O-ring
- Vespel/Graphite ferrules, when heated

Symptoms of leaks

- Higher than normal vacuum manifold pressure or foreline pressure
- Higher than normal background
- Peaks characteristic of air (m/z 18, 28, 32, and 44 or m/z 14 and 16)
- Poor sensitivity
- Low relative abundance of m/z 502 (this varies with the tune program and MSD used)

Remedy

- Check interface nut for tightness. Replace if necessary.
- Check and leak test the GC injection port.

MS interface column nut, 05988-20066

Universal column nut, 5181-8830

Replacement Parts

Description	Part No.
MS interface column nut, female	05988-20066
Column nut for long or long two-hole ferrules	05921-21170
Universal column nut, 2/pk	5181-8830

The easiest way to insure that you minimize background contamination and remove damaging oxygen from your carrier gas system is to use a carrier gas purifying trap right before the gas enters your GC system.

Cleaning Solvents

It is common to see cleaning solvent peaks in the mass spectra shortly after the ion source is cleaned.

Remedy

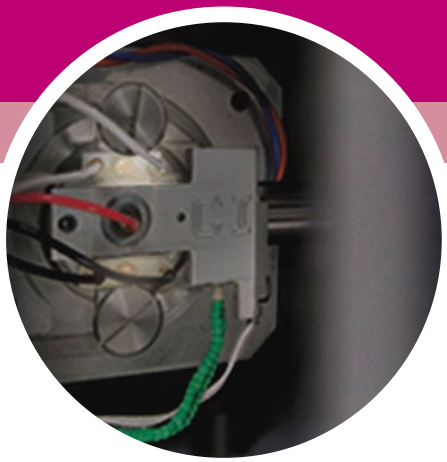
- Dry all cleaned metal parts in the GC oven before reassembling and reinstalling them. Refer to specific cleaning procedures in your MSD Hardware Manual or MSD Maintenance and Troubleshooting Manual.
- Use a temperature above the boiling point of the solvent but below the limit of the column.

Fingerprints

Fingerprints contain hydrocarbons that can appear in mass spectra. Hydrocarbon contamination is characterized by a series of mass peaks 14 m/z apart. The abundances of these peaks decrease as peak mass increases. Fingerprint contamination is usually caused by the failure to wear lint-free, nylon gloves during ion source cleaning, GC inlet maintenance, or from installing the column. Use special care to avoid recontamination of parts after you clean them. This typically occurs after some maintenance or part replacement.

Remedy

Reclean using clean, nylon gloves and proper cleaning techniques.



Diffusion Pump Fluid

If the diffusion pump fluid is allowed to operate with no column (carrier gas) flow into the vacuum system, vapor from the diffusion pump fluid can drift up into the vacuum manifold. A more serious problem is when fluid is back streamed into the vacuum manifold by sudden or improper venting of the vacuum system. If a diffusion pump has back streamed, a prominent peak will often be seen at m/z 446 and the spectral baseline will exhibit increased background noise.

Remedy

If m/z 446 appears, please call Agilent for assistance.

Foreline Pump Oil

Foreline pump oil contamination is characterized by peaks spaced 14 amu apart (hydrocarbons). Contamination with foreline pump oil is less common than contamination with diffusion pump fluid.

Remedy

Call Agilent for assistance.

A crude sign of a "leak-free" MS system is when the ion ratio of m/z 28 (nitrogen) over m/z 32 (oxygen) is approximately two or greater.

Even preconditioned ferrules can shrink slightly at very high temperatures, so if leak problems persist upon a new column installation, check this fitting first.

MSD Contamination Identification

The following table lists some of the more common contaminants, the ion characteristics of those contaminants, and the likely sources of those contaminants.

Common Contaminants

Ions (m/z)	Compound	Possible Source
13, 14, 15, 16	Methane	CI gas
18, 28, 32, 44 or 14, 16	H_20, N_2, O_2, CO_2, CO_2 or N, O	Residual air and water, air leaks, outgassing from Vespel ferrules
31, 51, 69, 100, 119, 131, 169, 181, 214, 219, 264, 376, 414, 426, 464, 502, 576, 614	PFTBA and related ions	PFTBA (tuning compound)
31	Methanol	Cleaning solvent
43, 58	Acetone	Cleaning solvent
78	Benzene	Cleaning solvent
91, 92	Toluene or xylene	Cleaning solvent
105, 106	Xylene	Cleaning solvent
151, 153	Trichloroethane	Cleaning solvent
69	Foreline pump fluid or PFTBA	Foreline pump oil vapor or calibration valve leak
73, 147, 207, 221, 281, 295, 355, 429	Dimethylpolysiloxane	Septum bleed or methyl silicone column coating
77, 94, 115, 141, 168, 170, 262, 354, 446	Diffusion pump fluid	Diffusion pump fluid and related ions
149	Plasticizer (phthalates)	Vacuum seals (O-rings) damaged by high temperatures, use of vinyl or plastic gloves
Peaks spaced 14 amu apart	Hydrocarbons	Fingerprints, foreline pump oil

Mass Spectrometer Symptoms

Mass spectrometer symptoms can typically be classified as either affecting system sensitivity or affecting the repeatability of a measurement. Most symptoms can be corrected by following the suggested corrective actions.

Mass Spectrometer Symptoms

Symptoms	Remedy
Sensitivity	
Wrong retention time	Check GC, method, application and carrier gas velocity.
Low signal	Check GC, tune vacuum system.
Leaking injection port	Clean the injection port. Replace the injection port liner and septa.
Air leak	Check and tighten interface nut, leak test GC injection port.
Peak widths	Do Autotune, check flow rate and temperature stability.
Interfering peaks	Check time parameters, coeluting peaks, column type.
Excessive background	Do Autotune and compare to background specifications. Check time parameters.
Incorrect mass assignment	Retune.
Abnormal spectra – excessive background contamination	Check for contamination.
Incorrect tuning	Check tune file, retune, check sample.
Repeller voltage is too low	Raise voltage to test for response.
Dirty ion source	Clean source.

5975 MSD

Mass Spectrometer Symptoms

Symptoms	Remedy
Repeatability	
Dirty syringe needle	Clean or replace the syringe.
Wrong syringe needle	Replace syringe and septa.
Leaking injection port	Perform injection port maintenance. Replace the injection port liner, septa and liner O-ring.
Injection is too large	Check method and injection volume, split ratio and/or splitless purge time.
Loose column connections	Tighten column nuts on injection port or transfer line. Replace column nuts and ferrule.
Variations in pressure, column flow and temperature	Ensure the MSD is located in an environment where the temperature is stable. Keep MSD out of drafts and direct sunlight. Check that the carrier gas is steady and well regulated. Service the foreline pump and/or diffusion pump.
Dirty ion source	Clean source.
Loose connections in the analyzer	Check internal and external analyzer wiring conections, make sure all are secure.
Ground loops	Check main electrical lines.

Cleaning and Maintenance Supplies

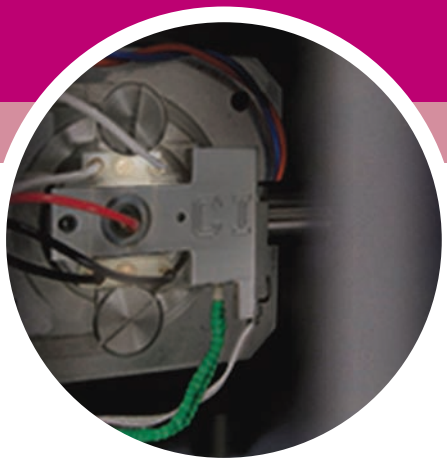
Description	Part No.
One Year Maintenance Kit (for diffusion pump systems) Includes Big Universal Trap for He (1/8 in.), abrasive sheets (5/pk), lint-free cloths (15/pk), cotton swabs (100/pk), SantoVac Ultra, 18.5 mL (2 each), rough pump oil (1 liter), filament assembly, Octafluoronapthalene (OFN)	5183-2096
Nylon gloves, lint-free, large, 1 pair	8650-0030
Nylon gloves, lint-free, small, 1 pair	8650-0029
Lint-free industrial wipes, 100% cotton, 9 x 9 in., 300/pk	9310-4828
Ion source cleaning kit Includes lint-free cloths (15/pk), abrasive sheets (5/pk), cotton swabs (100/pk), lint-free nylon gloves, Alumina powder, abrasive	5181-8863
Cloths, lint free, 15/pk	05980-60051
Cotton swabs, 100/pk	5080-5400
Abrasive sheets, aluminum oxide green lapping paper, 600 mesh, 5/pk	5061-5896
Alumina powder, abrasive, 1 kg	8660-0791
PFTBA sample, certified, 10 g	8500-0656
Replacement glass bulb for PFTBA and PFDTD test sample, 5975	G3170-80002
Replacement glass vial for PFTBA and PFDTD test sample	05980-20018
Activated alumina, absorbent pellets for Edwards rough pump traps, non-LC/MS, 1 lb can	8500-1233
MSD Tool Kit, 5975/5973 Includes source hold tool, lint-free cloth, cotton swabs, lint-free nylon gloves, abrasive sheets, wrenches and driving tools	G1099-60566
MSD Tool Kit, 5972/5971 Includes small cleaning rod, large cleaning rod, source hold tool, cotton swabs, lint-free nylon gloves, abrasive sheets, wrenches and driving tools	05971-60561

Column installation tool, G1099-20030

Vespel/Graphite ferrules, 5181-3323

Cleaning and Maintenance Supplies

Description	Part No.
Tools	
Screwdriver, 3 in. Pozidriv shaft No. 1 pt, fits no. 2-4 screws	8710-0899
Screwdriver, 4 in. Pozidriv shaft No. 2 pt, Fits no. 5-10 screws	8710-0900
Open end wrench, 1/4 and 5/16 in.	8710-0510
MS Interface Column Installation Tool	G1099-20030
Hex nut driver, 5.5 mm	8710-1220
Screwdriver, Torx T20	8710-1615
Screwdriver, Torx T15	8710-1622
Screwdriver, Torx T10	5182-3466
Ferrules	
0.4 mm Vespel Graphite ferrule for 200/250 µm columns, 10/pk	5062-3508
0.5 mm Vespel Graphite ferrule for 320 µm columns, 10/pk	5062-3506
250 µm Vespel/Graphite ferrule, 10/pk	5181-3323
SilTite metal ferrules for 1/16 in. OD tubing, 10/pk Includes 2 column nuts	5184-3571
SilTite Metal Ferrules, 1/16 in. x 0.4 mm ID, 10/pk Includes 2 column nuts	5184-3569
SilTite Metal Ferrules, 1/16 in. x 0.5 mm ID, 10/pk Includes 2 column nuts	5184-3570
MS Interface Supplies	
MS interface column nut, female	05988-20066
Column nut for long or long two-hole ferrules	05921-21170
Universal column nut, 2/pk	5181-8830



Ion Source

The ion source operates by electron ionization (EI) or chemical ionization (CI). The sample enters the ion source from the GC/MSD interface. Electrons emitted by a filament enter the ionization chamber, guided by a magnetic field. The high-energy electrons interact with the sample molecules, ionizing and fragmenting them. The positive voltage on the repeller pushes the positive ions into the lens stack, where they pass through several electrostatic lenses. These lenses concentrate the ions into a tight beam, which is directed into the mass filter.

Maintaining the Ion Source

Cleaning procedures for MSDs vary. Refer to your Troubleshooting and Maintenance manual for specific ion source cleaning procedures.

Common Measures of Instrument Performance

- Abundance of certain ions (e.g., percentage of the 502 ion from the Autotune report).
- Shape of lens ramps and the chosen voltages, especially Repeller Ramp.
- Sensitivity obtainable for a given analysis.
- Ability to tune to a given reference compound (e.g., DFTPP).

Electron Impact (EI) Ion Source

Preparing to Clean

Prior to cleaning, the mass spectrometer must be vented and the ion source must be removed. Before venting the system, the following conditions must be met:

- Heated zones are less than 100°C.
- The diffusion pump is off and cool.
- The turbo pump is off and not spinning.
- The rough pump is off.

Always allow the automatic venting routine to run its full course. Improper venting may cause diffusion pump fluid to be deposited into the analyzer (backstreaming). It can also reduce the life of the multiplier or other sensitive MS parts.

MSD Flowrates (mL/min)

	Min	Max Diff Pump	Max Turbo Pump	Tuning Max
5975	0.1	2.0	4.0	2.0
5973	0.1	2.0	4.0	2.0
5972	0.1	2.0	N/A	2.0
5971	0.1	1.5	N/A	1.0
GCD	0.1	1.0	N/A	1.0

Electron Impact (EI) Ion Source

The recommended cleaning material for the EI ion source is abrasive powder, aluminum oxide powder.

Abrasively clean the surfaces that contact the sample or ion beam. Use an abrasive slurry of alumina powder and reagent-grade methanol on a cotton swab. Use enough force to remove all discolorations. Polishing the parts is not necessary; small scratches will not harm performance. Also abrasively clean the discolorations where electrons from the filaments enter the source body.

Warnings & Caution

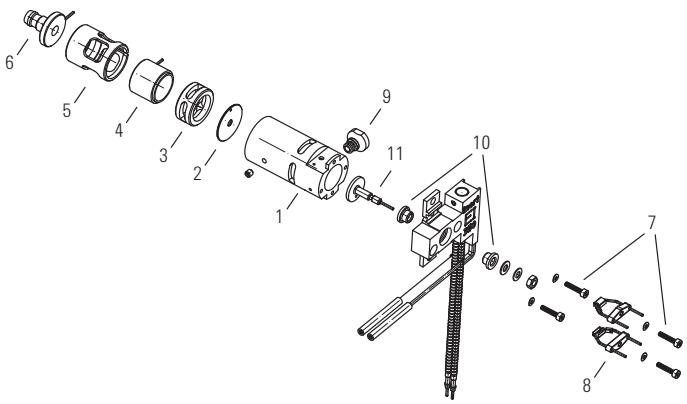
Important: Do not abrasively or ultrasonically clean the insulators.

Do not immerse filaments or lens insulators in solvent. If insulators are dirty, clean them with a cotton swab dampened with reagent-grade methanol. If that does not clean the insulators, replace them.

Take care to avoid contaminating cleaned and dried parts. Put on new, clean gloves before handling the parts. Do not set the cleaned parts on a dirty surface. Set them only on clean, lint-free cloths.

5975/5973 MSD Electron Impact Ion Source Parts (EI)

Item	Description	Part No.	Inert Part No.
1	Ion source body	G1099-20130	G2589-20043
2	Drawout plate, 3 mm	05971-20134	G2589-20100
3	Drawout cylinder	G1072-20008	G1072-20008
4	Lens insulator	G3170-20530	G3170-20530
5	Ion focus lens	05971-20143	05971-20143
6	Entrance lens	G3170-20126	G3170-20126
7	Cap screw, gold plated	G1999-20021	G1999-20021
8	High temperature filament	G2590-60053	G2590-60053
9	Transfer line socket	G1099-20136	G1099-20136
10	Repeller insulator	G1099-20133	G1099-20133
11	Repeller	G1099-20132	G2589-20044



5975/5973 MSD Electron Impact (EI) ion source assembly

Electron Impact (EI) Ion Source

5972/5971/GCD MSD Ion Source Parts (EI)

Description	Part No.
Entrance lens	05971-20126
Lens insulator, 597X MSD	G3170-20530
Ion focus lens	05971-20143
Drawout cylinder	G1072-20008
Drawout plate, 3 mm	05971-20134
Set screw	0515-1446
Repeller assembly	05971-60170
Screw for filament on the source	0515-1046
Transfer line tip, gold plated, 5972/5971	05971-20305

Tips & Tools

It is good practice to replace scratched lenses and other ion source parts. Scratched source parts lead to poor performance.

Chemical Ionization (CI) Ion Source

Because the CI ion source operates at much higher pressures than the EI ion source, it will probably require more frequent cleaning than the EI ion source.

The source should be cleaned whenever there are performance anomalies that are associated with a dirty ion source. Let analytical performance be your guide.

When cleaning the CI ion source, concentrate on the CI repeller, ion source body, and draw out plate. Be sure to clean the 0.5 mm diameter holes in the ion source body and draw out plate.

Cleaning the ion source is very similar to cleaning the EI ion source. Use the same EI cleaning procedure with the following exceptions:

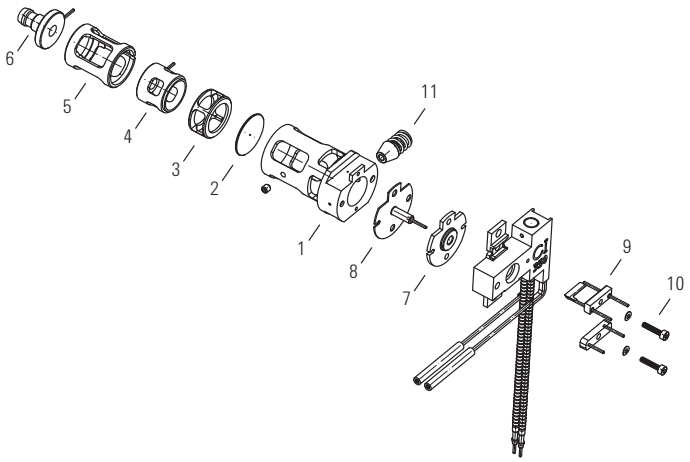
- The CI ion source may not look dirty but deposits left by chemical ionization are very difficult to remove. Clean the CI ion source thoroughly.
- Use a round wooden toothpick to gently clean out the electron entrance hole in the source body and the ion exit hole in the draw out plate.
- Do not use halogenated solvents. Use hexane for the final rinse.

Tips & Tools

Visual appearance is not an accurate guide to cleanliness of the CI ion source. The CI ion source can show little or no discoloration yet still need cleaning.

5975/5973 MSD Chemical Ionization Ion Source Parts (CI)

Item	Description	Part No.
1	Source body	G1999-20430
2	Draw out plate	G1999-20446
3	Draw out cylinder	G1999-20444
4	Lens insulator	G3170-20540
5	Ion focus lens	G1999-20443
6	Entrance lens	G3170-20126
7	Repeller insulator	G1999-20433
8	Repeller	G1999-20432
9	High temperature filament	G1099-80053
10	Cap screw, gold plated	G1099-20021
11	Interface tip seal/spring	G3170-60412



5975/5973 MSD Chemical Ionization (CI) ion source assembly

Installing a Capillary Column in the GC/MSD Interface

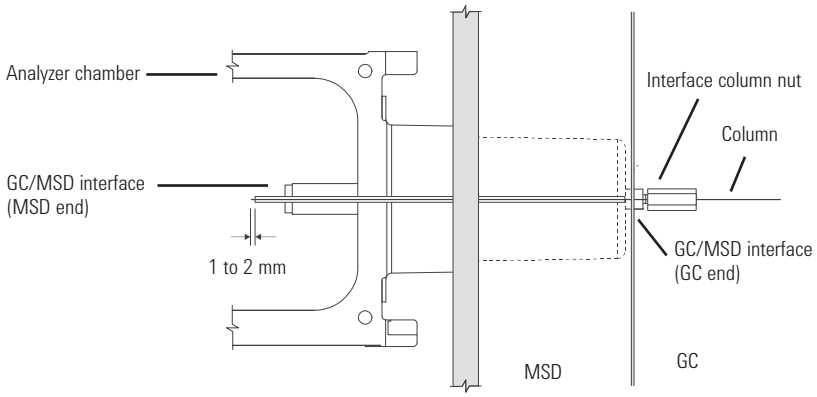
1. Condition the column.
2. Vent the MSD and open the analyzer chamber. Be sure you can see the end of the GC/MSD interface.
3. If the CI interface is installed, remove the spring-loaded tip seal from the MSD end of the interface.
4. Slide an interface nut and conditioned ferrule onto the free end of the GC column. The tapered end of the ferrule must point towards the nut.
5. Slide the column into the GC/MSD interface until you can pull it out through the analyzer chamber.
6. Break 1 cm off the end of the column. Do not let any column fragments fall into the analyzer chamber. They could damage the turbo pump.
7. Clean the outside of the free end of the column with a lint-free cloth moistened with methanol.
8. Adjust the column.
   - 5975 – Push the column through, and then let it pass the end of the Transferline by 1-2 mm. With the analyzer door partially open, view through the glass plate to see the column protrude.
   - 5973 – Push the column through, and then let it pass the end of the Transferline by 1-2 mm as seen with the analyzer door open from that side.
   - 5972 – Push the column in all the way and then pull it back about 1-2 mm.

   Use the flashlight and magnifying glass if necessary to see the end of the column inside the analyzer changer. Do not use your finger to feel for the column end.
9. Hand-tighten the nut. Make sure the position of the column does not change as you tighten the nut. Reinstall the spring-loaded tip seal if it was removed earlier.
10. Check the GC oven to be sure that the column does not touch the oven walls.
11. Tighten the nut 1/4 to 1/2 turn. Check the tightness after one or two heat cycles.

Tips & Tools

The column installation procedure for the 5975 MSDs is different from that for most previous MSDs. Using the procedure from another instrument may result in poor sensitivity and possible damage to the MSD.

Installing a capillary column in the GC/MSD interface



QuickSwap restrictor

QuickSwap MS Interface Restrictors

Agilent's QuickSwap capillary flow technology module and pre-swaged fused silica tubing restrictors can increase the productivity of your Agilent 5973N and 5975 inert MSD systems, allowing you to change columns without venting the MSD. QuickSwap not included.

These restrictors are prefabricated for convenience and ease of use. For applications requiring other restrictor sizes, Agilent offers a wide variety of deactivated fused silica tubing, SilTite ferrules and swaging tools.

QuickSwap MS Interface Restrictors

Description	ID (mm)	Unit	Part No.
Quick Swap Restrictor	0.092	4/pk	G3185-60361
Quick Swap Restrictor	0.100	4/pk	G3185-60362
Quick Swap Restrictor	0.110	4/pk	G3185-60363
Quick Swap Restrictor	0.120	4/pk	G3185-60364
Quick Swap Restrictor Variety Pack, 2 each of the above ID restrictors			G3185-60300

MSD Filaments

Like the filaments in an incandescent light bulb, the ion source filaments will eventually burn out. Certain practices will reduce the chance of early failure.

- When setting up data acquisition parameters, set the solvent delay so that the analyzer will not turn on while the solvent peak is eluting.
- When the software prompts 'Override solvent delay at the beginning of a run' always select 'No.'
- Higher emission current will reduce filament life.
- If you are controlling your MSD from the Edit Parameters screen, always select 'MS Off' before changing any of the filament parameters.

EI high temperature filament, G2590-60053

MSD Filaments

Description	5975 Series	5973 Series	5972 Series	5971 Series
Filament assembly (EI)	G2590-60053	G2590-60053	G2590-60053	05971-60140
Filament assembly (CI)	G1099-80053	G1099-80053		
Micro ion vacuum gauge	G3170-80001			
Triode gauge tube for measuring vacuum		0960-0897		

Tips & Tools

It is very useful to switch from one filament to the other every three months so that when filament fails, you know the other will fail soon. This will allow you to change both filaments at the same time. Since the GC/MS system is already vented, it's a good idea to replace other supplies in the flowpath at the same time as the filaments.

Quadrupole Mass Filter

The mass filter does not require periodic maintenance. It should not be removed from the radiator or distributed in any way.

- Never put the quadrupole in an ultrasonic cleaner.
- Never change the physical orientation of the quadrupole mass filter.
- The fused-quartz quadrupole is fragile and will break if dropped or handle roughly.
- The material in the cusps of the quadrupole is very hygroscopic. If exposed to water, the quadrupole must be dried very slowly to prevent damage.
- Cleaning techniques appropriate for other manufacturers' instruments are not suitable for Agilent MSDs – and may actually harm the mass filter.
- To save time and effort, use only Agilent MSD mass filters, which do not require periodic cleaning or maintenance.
- In case of extreme contamination, contact a trained Agilent service representative to perform the mass filter cleaning.

Vacuum Systems and Pumps

The vacuum system creates the high vacuum (low pressure) required for the MSD to operate. Without this vacuum, the molecular mean free path is too short.

Ions cannot travel from the ion source through the mass filter to the electron multiplier (detector) without colliding with other molecules.

The main components of the vacuum system are:

- Vacuum manifold
- Foreline gauge
- Calibration valve
- Gauge controller (optional)
- Vacuum seals
- Foreline pump and/or trap
- Diffusion/turbo pump and fan
- High vacuum gauge tube

Tips & Tools

Keeping a pan under the vacuum pump helps to detect and identify the origin of oil leaks.

Pressure Symptoms

This section describes unusual pressure readings and their possible causes. The symptoms in this section are based on typical pressures. At typical column flow rates (0.5 - 2.0 mL/min), the foreline pressure will be approximately 20 to 100 mTorr. The vacuum manifold pressure will be approximately 1 x 10^{-6} to 1.4 x 10^{-4} Torr.

These pressures can vary widely from instrument to instrument, so it is important that you are familiar with the pressures that are typical for your instrument at a given carrier gas flow and oven temperature.

The foreline pressures listed can only be measured on diffusion pump-equipped systems. Turbomolecular pumps are controlled according to their speed and do not have foreline pressure gauges.

The vacuum manifold pressures can only be measured if your system is equipped with the optional gauge controller.

Pressure Symptoms

Symptoms	Possible Causes
Foreline pressure is too high.	
• Pressure is above 100 mTorr. • Pressure for a given column flow has increased over time.	• Column (carrier gas) flow is too high. • Wrong carrier gas. • Air leak (normally at transferline interface). • Foreline pump oil level is low or oil is contaminated. • Foreline hose is constricted. • Foreline gauge is not working correctly. • Foreline pump is not working correctly.
Foreline pressure is too low.	
• Pressure is below 20 mTorr.	• Column (carrier gas) flow is too low. • Wrong carrier gas. • Column plugged or crushed by an overtightened nut. • Empty or insufficient carrier gas supply. • Bent or pinched carrier gas tubing. • Foreline gauge is not working correctly.
Vacuum manifold pressure is too high.	
• Pressure is above 1.4 x 10^{-4} Torr. • Pressure for a given column flow has increased over time.	• Column (carrier gas) flow is too high. • Wrong carrier gas. • Air leak. • Foreline pump is not working correctly. • Diffusion pump fluid level is low or fluid is contaminated. • Defective gauge controller. • Faulty ion gauge tube.
Vacuum manifold pressure is too low.	
• Pressure is below 1.4 x 10^{-4} Torr.	• Column (carrier gas) flow is too low. • Wrong carrier gas. • Column plugged or crushed by an overtightened nut. • Empty or insufficient carrier gas supply • Bent or pinched carrier gas tubing. • Defective gauge controller. • Faulty ion gauge tube.

Diffusion Pump

It is not necessary to change the diffusion pump fluid more than once a year, unless you observe symptoms that suggest a problem with the diffusion pump fluid. The MSD must be vented in order to check the diffusion pump fluid (except for the 5975/5973). Therefore, the best time to check the fluid is when the instrument is already vented for other maintenance.

How to Check the Fluid Level

5972/5971 Series

- If it is not vented already, shut down and vent the MSD according to instrument manual.
- Unplug the MSD power cord.
- Remove the pump and cover the top with aluminum foil.
- After heating the pump in a GC oven at 60°C for 15 minutes to make the fluid flow down into the reservoir at the bottom, remove the stack parts.
- Inspect the pump fluid. If the fluid is discolored or contains particulate material, the fluid must be changed.
- Use a metal ruler to determine the depth of the fluid. A pump that has been in operation should have a pool 9 mm +/- 1 mm deep. Fluid in freshly charged pumps will be 12 mm deep. It is normal that up to 2 mL of oil may be in the rear portion of the vacuum manifold. The recommended total fluid charge for the 5971/5972 is 18 mL (+/- 2 mL).

5975/5973 Series

- Use the sight glass to determine the depth of the fluid. The recommended total fluid charge is approximately 37 mL.

Foreline Pump

Foreline Pump

The oil in the foreline or rough pump should be replaced on average once every six months, but can vary depending upon applications. After oil replacement, if a foreline trap is present, the molecular sieves should be replaced.

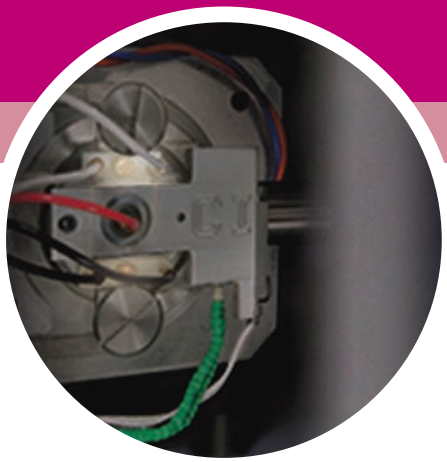
Avoid contact with the pump oil. The residue from some samples may be toxic. Dispense of used oil properly.

Pump Oils

Description	5975 Series	5973 Series	5972/5971 Series
Diffusion pump fluid, 18.5 mL	6040-0809 2 required	6040-0809 2 required	6040-0809
Foreline pump oil, P3, 0.5 L	6040-0621		
Foreline pump oil, Inland 45, 1 L		6040-0834	6040-0834
High vacuum grease, 25 g	6040-0289	6040-0289	6040-0834

General Instructions on How to Replace the Pump Oil

- Vent and shut down the MSD.
- Place a container under the drain plug on the foreline pump.
- Remove the fill cap from the top of the pump to expose the fill hole.
- Remove the drain plug from the pump.
- Reconnect the MSD to its power source. Switch on for 2 or 3 seconds, and then switch it off again. This displaces old oil from the internal pump cavities. Disconnect the power cord again.
- Reinstall the drain plug and pour pump oil into the fill hole.
- Reinstall the fill cap.
- Reconnect the MSD power cord.
- Start up and pump down the MSD according to the Instrument Manual procedure.



MSD Electron Multipliers and Replacement Horn

The lifetime of an electron multiplier is directly related to the current that flows through it and the extent of contamination or condensation that it experiences. Replace the electron multiplier or replacement horn when voltage is over 2500 volts. To maximize electron multiplier life:

- Maintain the best possible vacuum, especially in the analyzer manifold.
- Use extreme caution and be conservative with venting, pumpdown, and all vacuum system procedures to keep pump fluid background to a minimum.
- After venting, allow four hours for pumpdown and thermal equilibration before scanning.
- Actively look for background contamination and leaks and repair them immediately.
- Don't tune excessively. PFTBA can result in higher background over an extended period of time.
- Replace the electron multiplier if vacuum is poor or voltage is over 2500 volts.

Tips & Tools

These are the recommended replacement multipliers and horns for the MSD. Other manufacturers' products may be incompatible with Agilent instruments and can result in reduced sensitivity, lifetime, and noise problems.

MSD Electron Multipliers and Replacement Horn

Description	5975 Series	5973 Series	5972/5971 Series
Electron multiplier replacement horn Use with electron multipliers with "straight" horns.	05971-80103	05971-80103	05971-80103
Triple axis detector assembly*	G3170-80100		
Triple axis electron multiplier	G3170-80103		
EM signal wire, low noise detector	G3170-80008		
High energy dynode		G1099-80001	
Electron multiplier			05971-80102

**Include on 5975 TAD systems*

MS standards

MS Test and Performance Samples

Tuning Samples			Performance Verification Samples			Checkout Samples		
MSD	**EI Tune**	**CI Tune**	**EI**	**Negative Mode CI**	**Positive Mode CI**	**HighMass**	**Semi-Volatile**	**Volatile**
5975	PFTBA	PFDTD	OFN 1 pg/µL	OFN 100 pg/µL	Benzophenone 100 pg/µL	PFHT	DFTPP	BFB
5973	PFTBA	PFDTD	OFN 1 pg/µL	OFN 1 pg/µL	Benzophenone 100 pg/µL		DFTPP	BFB
5972	PFTBA	PFTBA	HCB 10 pg/µL	N/A	Benzophenone 100 pg/µL		DFTPP	BFB
5971	PFTBA	PFTBA	Methyl Stearate 500 pg/µL	N/A	Benzophenone 100 pg/µL		DFTPP	BFB
GCD	PFTBA	N/A	Sample A 10 ng/µL	N/A	N/A		DFTPP	BFB

Tips & Tools

Each GC/MS has a specific test and performance sample. Refer to the chart for the exact sample. All volumes are approximately 0.5-1 mL unless otherwise specified.

Calibration

The calibration valve is an electromechanical valve with a vial for tuning compounds. Perfluorotributylamine (PFTBA) is the most commonly used tuning compound. It is required for automatic tuning of the MSD in EI mode. The tuning compound is usually a liquid but can be a volatile or semi-volatile solid.

The calibration vial can be refilled without venting the system. Fill the vial to 0.5 cm from the top. Do not overfill. Air is trapped in the vial when it is refilled. This sometimes causes an "Excess source pressure" error message during the first tune after refilling. This is more likely if the vial is overfilled. Be sure to purge the air upon refilling the vial.

Calibration Standards

Description	5975 Series	5973 Series	5972 Series	5971 Series
OFN, 1 pg/µL	5188-5348	5188-5348		
OFN, 100 fg/µL	5188-5347			
Benzophenone, 100 pg/µL	8500-5440	8500-5440	8500-5440	8500-5440
PHFT, 100 pg/µL	5188-5357			
PFTBA sample, certified, 10 g	8500-0656	8500-0656	8500-0656	8500-0656
PFTBA MS Sample Kit, 0.5 mL	05971-60571	05971-60571		
PFDTD calibrant	8500-8510	8500-8510		
Evaluation sample Solution of dodecane, biphenyl, p-cholorodiphenyl, and Methyl palmitate in isooctane. Six 1.0 mL ampoules: 4 at 10 ng/µL, 1 at 100 ng/µL, 1 at 100 pg/µL.	05970-60045	05970-60045	05970-60045	05970-60045
p-Bromofluorobenzene (BFB), 25 µg/mL	8500-5851	8500-5851	8500-5851	8500-5851
Hexachlorobenzene 10 pg/µL, 1 ng/µL			8500-5808	
Methyl stearate (in methanol); 1 ng/µL, 2 ea				05990-60075
GC/MS tuning standard	8500-5995	8500-5995	8500-5995	8500-5995

The Agilent J&W GC Column Selection Guide

Agilent J&W
GC Column Selection Guide
Rely on unsurpassed reproducibility, efficiency, and inertness.
Speed your selection with this one-stop resource.
Agilent Technologies

Your single source for the highest-performing GC columns, plus expert advice to help you achieve reliable results, faster.

From low-bleed columns for MS... to premium polysiloxane columns... to specialty columns for pesticide, petroleum, or life science applications... our GC Column Selection Guide has everything you need to analyze your most challenging compounds. You will also find...

- A review of GC theory, including retention time, separation factor, theoretical plates, resolution, and more.
- Strategies for increasing your productivity and speeding up the troubleshooting process.
- The most current method development procedures.
- Concise column selection flowcharts with industry-specific guidelines.

To request your copy of this concise, easy-to-use resource, go to **www.agilent.com/chem/guides**. Or ask your local Agilent Representative or Agilent Authorized Distributor for publication number 5989-6159EN.

How to contact Agilent

For the latest information on the complete line of Agilent Technologies columns and supplies:

- Visit our Web site: **www.agilent.com/chem**
- Contact your local Agilent sales office
- Contact your local Agilent Authorized Distributor

Information, descriptions, and specifications in this publication are subject to change without notice.

© Agilent Technologies, Inc. 2008
Printed in the USA April 14, 2008
5989-7612EN

Agilent Technologies